

Flow and fracture of austenitic stainless steels at cryogenic temperatures

P. Fernández-Pisón ^{*1,3}, J. A. Rodríguez-Martínez¹, E. García-Tabarés²,
I. Avilés-Santillana³, and S. Sgobba³

¹Department of Continuum Mechanics and Structural Analysis, University Carlos III of Madrid, Leganés, Madrid, Spain

²Department of Physics, University Carlos III of Madrid, Leganés, Madrid, Spain

³European Organization for Nuclear Research (CERN), Genève, Switzerland

Abstract

In this paper, we have characterized the microstructural evolution and the plastic flow and fracture behaviours of AISI 304L and AISI 316LN stainless steel grades at liquid nitrogen temperature (77 K) and at liquid helium temperature (4 K). Uninterrupted tensile experiments, where the sample is continuously deformed under quasi-static loading conditions until fracture, have been carried out with a Single-Section Sample to obtain the stress-strain characteristics of the two grades. Interrupted tensile experiments, in which the sample is unloaded before fracture, have been performed with a novel Double-Section Sample to later characterize the strain-induced martensitic transformation at different levels of deformation. The content of martensite has been determined post-mortem, using magnetic induction, electron backscatter diffraction and quantitative light optical micrography. The results obtained with the three methods show quantitative agreement, and reveal that the martensitic transformation in AISI 304L occurs faster and to a greater extent than in AISI 316LN both at 77 K and at 4 K. To the authors' knowledge, in this paper we provide the first experimental results for the evolution of the content of strain-induced martensite in AISI 304L and AISI 316LN samples tested at liquid helium temperature. In addition, the experimental data for the evolution of the martensite volume fraction with the strain have been used to identify the temperature-dependent parameters of the martensitic transformation kinetic model proposed by Olson and Cohen (1975). Moreover, Mode I fracture tests with fatigue-precracked Compact Samples have been carried out to determine the fracture properties of the two investigated materials using the "resistance curve procedure" (ASTM-E1820-20a, 2020). The crack-growth resistance curves have been obtained with four different methods here referred to as ASTM Compliance Method, W-N Compliance Method, Modified W-N Compliance Method and ASTM Normalization Method, which is an original methodological contribution of this paper. While the four approaches yield similar results for the fracture toughness, only the W-N Compliance Method and the Modified W-N Compliance Method, the latter being proposed in this paper, fulfil all the requirements of the standard ASTM-E1820-20a (2020) so that the calculated fracture toughness can be accepted as a material property. The comparison of results for both materials and testing temperatures shows that AISI 316LN displays higher fracture toughness than AISI 304L. Moreover, post-mortem microstructural analysis of the Compact Samples near the fracture surface has revealed that the content of martensite is greater in AISI 304L than in AISI 316LN. Furthermore, for AISI 304L more martensite is formed in the sample tested at 77 K because the plastic deformation near the crack is more than at 4 K.

*mariapilar.fernandez@uc3m.es

Nomenclature

Latin characters

a	crack size: distance from the load-line to the crack tip
a_0	initial crack size: distance from the load-line to the fatigue precrack tip
b_0	initial remaining ligament: distance from the fatigue precrack tip to the sample back edge ($W - a_0$)
B	thickness of the sample
C_i	constant parameters used in the definition of the power law regression line $J = C_1 \Delta a^{C_2}$ ($i=1,2$)
D	average grain diameter
$f^{\alpha'}$	volume fraction of α' -martensite
J	J -integral
J_{Ic}	plane-strain fracture toughness characterized by J -integral (size-independent J_Q)
J_Q	crack initiation toughness characterized by J -integral (at 0.2 mm of crack extension beyond blunting)
$J - R$ curve	crack extension resistance as a function of stable crack extension
K, K_I	stress intensity factor for Model-I crack
K_{Ic}	plane-strain fracture toughness characterized by K-factor
K_{JIc}	plane-strain fracture toughness characterized by K-factor converted from J_{Ic} (size-independent K_{JQ})
K_{JQ}	crack initiation toughness characterized by K-factor converted from J_Q
P	load
R	load ratio ($R = P_{min}/P_{max}$)
t	time
T	temperature
v	load-line displacement
W	width of the sample

Greek characters

α	body-centred cubic lattice (ferrite)
α'	body-centred tetragonal lattice (martensite)
γ	face-centred cubic lattice (austenite)
Δa	crack extension ($a - a_0$)
ϵ	hexagonal close-packed lattice (martensite)
ζ	parameter of the Olson and Cohen model: rate of shear-band formation
ζ_i	parameters to model the temperature-evolution of the Olson and Cohen model parameter ζ ($i = 1, 2, 3$)
ξ	parameter of the Olson and Cohen model: probability of a shear-band intersection forming a martensitic embryo
ξ_i	parameters to model the temperature-evolution of the Olson and Cohen model parameter ξ ($i = 1, 2, 3$)
σ	stress (Cauchy)
σ_{TS}	ultimate tensile strength
σ_Y	effective yield strength ($\sigma_Y = (\sigma_{YS} + \sigma_{TS})/2$)
σ_{YS}	yield strength (0.2% offset)
ϵ	strain (Hencky)
$\dot{\epsilon}$	initial strain rate (Hencky)

Acronyms

CICC	Cable-In-Conduit Conductors
------	-----------------------------

EBS	Electron Backscatter Diffraction
FEG	Field Emission Gun
LHC	Large Hadron Collider
SEM	Scanning Electron Microscope
W-N	Weiss-Nyilas (Compliance Method and Modified Compliance Method)

1 Introduction

Superconducting high-field magnets have become indispensable in high-energy particle physics accelerators to accurately guide the particle beams, such as at the LHC at CERN (Brüning et al., 2012), and in fusion reactors to confine and control the plasma, such as ITER (Mitchell et al., 2008).

The magnet systems, as they are used in CERN’s LHC or ITER, contain coils made of niobium-titanium (NbTi) or niobium-tin (Nb₃Sn) conductors, that need to be cooled to cryogenic temperatures to achieve the superconducting state (Brüning et al., 2012; Apollinari et al., 2015; Mitchell et al., 2008). Superconducting cables allow to apply very high currents that induce strong magnetic fields causing high magnetic forces within the magnet system. These forces have to be counteracted by structural components at the named cryogenic temperatures. Since austenitic stainless steels display stable material properties over the whole temperature operating range of the superconducting magnets, they are the dominant structural materials used for these components (Sgobba, 2006; Barabash et al., 2007). Such components are, for instance, the steel collars of the standard Dipole magnet for the LHC (Bertinelli et al., 2006) and the jacket of the so-called ITER “Cable-In-Conduit Conductors” (CICC) (Devred et al., 2014). The austenitic stainless steels used for the steel collars display at 4 K a yield strength, ultimate tensile stress and elongation higher than 1300 MPa, 1800 MPa and 24%, respectively (Bertinelli et al., 2006; Couturier and Sgobba, 2000); while in the case of the CICC jackets those properties are higher than 700 MPa, 1000 MPa and 20%, respectively (Romano et al., 2016). In both cases stress and deformation values are expressed using the engineering definition.

Austenitic stainless steels generally retain high strength, ductility and toughness at low temperatures, and they are paramagnetic or antiferromagnetic under the Néel temperature in the fully austenitic state. Nevertheless, they are susceptible to develop strain-induced martensitic transformation, especially at cryogenic temperatures. The initially undeformed γ -austenitic phase (face-centred cubic) may be transformed, by different transformation mechanisms, either to α' -martensite (body-centred tetragonal) or to ϵ -martensite (hexagonal close packed). After a relatively low deformation, the strain-induced α' -martensitic transformation is more extensive than the transformation of ϵ -martensite (Mangonon and Thomas, 1970; Seetharaman, 1984; De et al., 2004). Moreover, the α' -martensitic phase is ferromagnetic and becomes brittle at cryogenic temperatures. For these reasons, the γ - α' transformation is a major concern due to the high impact on the performance of the structural components used in high-field magnet systems. A comprehensive analysis of the flow and fracture behaviours of austenitic stainless steels at low temperatures, as applied for the superconducting magnets used at CERN and ITER, is therefore essential.

The manuscript is organized as follows. The austenitic stainless steels AISI 304L and AISI 316LN investigated in this work are presented in Section 2, including their chemical composition and the analysis of their microstructure in the as-received condition. A detailed description of the experimental techniques employed to characterize the tensile flow behaviour and microstructural evolution of AISI 304L and AISI 316LN at 300 K, 77 K and 4 K is provided in Section 3, and the analysis of results is performed in Section 4. Moreover, Section 5 describes the Compact Sample used to perform the fracture toughness tests, the four methods employed to construct the $J-R$ crack-growth resistance curves and the optical microscopy technique used to determine the strain-induced martensite formed near the fracture surface during the experiments, while

the results of the fracture tests are presented and discussed in Section 6. A summary of the research including the main findings derived from this investigation is given in Section 7.

2 Materials

The materials studied are AISI 304L and AISI 316LN austenitic stainless steels. AISI 304L is a general-purpose grade, while AISI 316LN is selected for applications requiring high strength, or where an increase of magnetic susceptibility might be of concern (Vogt et al., 1991). AISI 304L is the low (L) carbon version of the common iron-chromium-nickel AISI 304. On the other hand, AISI 316LN is a modification of the molybdenum-containing grade AISI 316, with low (L) content of carbon and addition of nitrogen (N) (Reed, 1989). The effect of molybdenum is the improvement of corrosion resistance and a higher stability of the austenite against martensitic transformation. Nitrogen also increases austenite stability, and enhances the formation of austenite compared to ferrite. Moreover, nitrogen substantially increases strength and maintains ductility down to cryogenic temperatures (Sgobba, 2006). The specific chemical compositions of the AISI 304L and AISI 316LN grades investigated in this work are given in Table 1. Both grades show a high purity which is a requirement for products intended for high vacuum purposes at CERN. Special attention is taken in reducing the content of sulphur and phosphorus in order to minimize the risk of microfissuring in austenitic stainless steel welds (Kujanpaa et al., 1979; Sgobba, 2006). Thus, while AISI 304L is melted in an electric arc furnace followed by secondary metallurgy (vacuum oxygen decarburization), the manufacturing process of AISI 316LN includes an electro-slag remelting which allows to keep the sulphur levels in the ppm range (Chakrabarti, 2006).

Grade	C	S	N	Cr	Ni	Mn	Si	Mo	P	Co
304L	0.013	0.0003	—	18.19	10.28	1.27	0.45	—	0.024	0.024
316LN	0.009	<0.002	0.19	17.37	13.09	1.34	0.28	2.61	0.024	0.100

Table 1: Chemical composition of AISI 304L and AISI 316LN (product analysis except for Co 316LN which is heat analysis, weight-% Fe balance).

The raw materials were provided in the form of hot rolled and solution annealed plates of 12 mm thickness (surface finish 1D according to the standard EN-10088-2 (2008) as indicated in the corresponding CERN technical specifications). For AISI 304L the annealing was performed at 1060 °C for 18 min followed by water quench, while for AISI 316LN it was done at 1110 °C for 9 min also followed by water quench. Fig. 1 shows the undeformed microstructure of both materials. Rolling direction is horizontal in the micrographs. AISI 304L presents some linear α -ferrite (body-centred cubic) precipitations embedded in the austenitic matrix along the rolling direction. The ferritic phase has been identified with the selective Murakami’s etchant as indicated in the standard ASTM-E407-07 (2015). Indeed, according to Sgobba (2006), AISI 304L grade normally retains some ferrite when cooled from solution annealing. Measurements with the Feritscope confirm that the ferrite volume is lower than 0.5% (see the item **Magnetic induction** in Section 3.2). On the other hand, AISI 316LN is constituted of 100% austenite, which is expected due to the strong austenite stability provided by the alloying elements. Moreover, the microstructure of both grades, AISI 304L and AISI 316LN, shows some annealing twins which are typically formed in austenitic stainless steels after deformation and subsequent annealing (Jin et al., 2013; Singh, 1996). The average grain size in the undeformed state is $D = 89.9 \mu\text{m}$ for AISI 304L and $D = 53.4 \mu\text{m}$ for AISI 316LN. These values were provided by the corresponding materials’ certificates, which determined the average grain size according to the standard ASTM-E112-13 (2013).

The thermal stability of AISI 316LN was confirmed by immersion in liquid helium for more

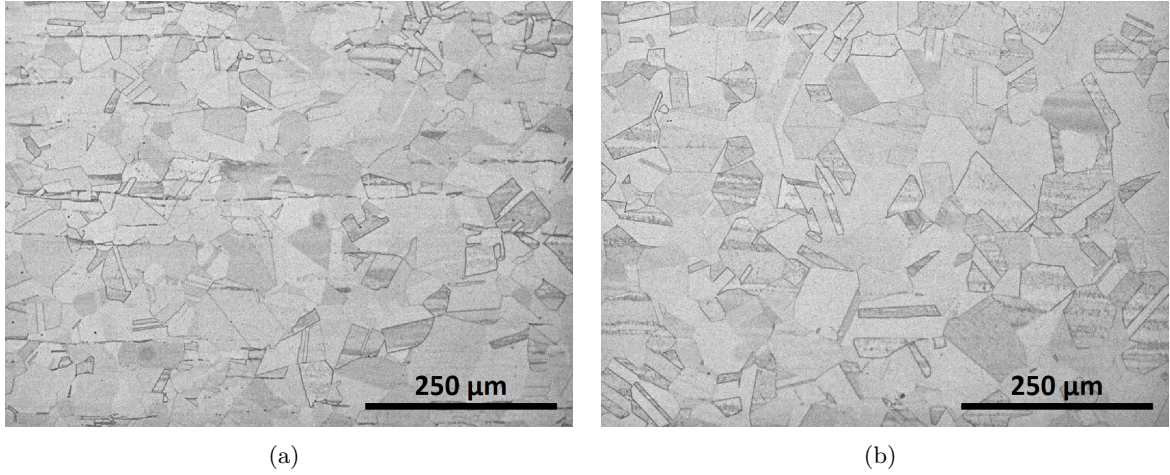


Figure 1: Light optical microscopy images of the materials' cross-section (through the plate's thickness, along the rolling direction). Rolling direction is horizontal in the micrographs. Images taken after oxalic acid etching (etchant no. 13 in ASTM-E407-07 (2015)): (a) AISI 304L and (b) AISI 316LN.

than two hours, after which no ferromagnetic response was detected with the Feritscope thus ruling out a spontaneous martensitic transformation. Regarding AISI 304L, there was not an evident increase of the material's ferromagnetic response neither after immersion in liquid nitrogen nor in liquid helium. Therefore, the spontaneous transformation of AISI 304L, if any, is negligible.

3 Flow behaviour: experimental techniques

3.1 Mechanical tests

Uniaxial tensile tests have been performed at room temperature ($T = 300$ K), at liquid nitrogen temperature ($T = 77$ K) and at liquid helium temperature ($T = 4$ K), according to the standards ISO-6892-1 (2009), ISO-6892-3 (2015) and ISO-6892-4 (2015), respectively. All the tests have been carried out at CERN in a 100 kN servo-hydraulic testing machine fabricated by MICROTEST. Two Epsilon cryogenic extensometers (3442C-050M-020-LT) have been employed for the tests in their shortest configuration of 12.5 mm gauge length which maximises the available strain measuring range. All the tests have been performed under displacement control of the machine's cross head at constant speed. For the tests performed at room temperature the samples have been directly attached to the machine's cross head, whereas for the cryogenic tests the in-house developed cryostat described by Avilés-Santillana et al. (2015) and Malik (2021) has been employed. The tensile samples have been extracted from the mid-thickness region of each plate with the tensile axis parallel to the rolling direction ("longitudinal flat tension test" according to the standard ASTM-A370-15 (2015)). The thickness of the samples is 4 mm. Two different geometries, here referred to as Single-Section Sample and Double-Section Sample, have been used:

- **Single-Section Sample:**

It has been used in uninterrupted tensile tests where the sample is continuously deformed until fracture. The aim is to obtain the complete stress-strain curve of the material. The design of the Single-Section Sample (Fig. 2) is based on a "non-proportional test piece" according to the standard ISO-6892-4 (2015). For each material, two uninterrupted tensile experiments have been performed for the three different testing temperatures.

For all performed tensile tests, the stress has been computed using the load values from the machine’s load cell (see Fig. 4). The strain values have been calculated using the displacement obtained from the extensometers: a single extensometer in the case of a Single-Section Sample, and two extensometers in the case of a Double-Section Sample.

3.2 Martensite measurements

We have used three complementary methods to determine the martensite volume fraction in the tested specimens (post-mortem measurements): magnetic induction, electron backscatter diffraction (EBSD) and quantitative light optical micrography. All the measurements have been performed at CERN. The idea is to compare the results obtained with the three approaches and provide consistent data for the evolution of the α' -martensite content with the strain for different testing temperatures. Note that Cao and Iwamoto (2017) and Cao et al. (2020) have recently determined the relative magnetic permeability of austenitic stainless steel samples during tensile testing at room temperature at different strain rates, thus obtaining a continuous measurement of the evolution of α' -martensite during loading. However, the application of this approach to tests performed at cryogenic temperatures is still pending.

- **Magnetic induction**

The Feritscope locally determines the content of the ferromagnetic phase in the material using a magnetic induction probe. The Feritscope Fisher model FMP30 employed in this study was supplied with certified ferrite standards which were used for the calibration, and therefore the device provides the measurements in ferrite percent independently of the nature of the ferromagnetic phase. Moreover, according to the Feritscope manual (FERITSCOPE-FMP30, 2008), measurement readings taken at a distance less than 2 mm from the edge of the sample have to be corrected by provided correction curves. Specifically, the correction is done by multiplying the device measurements by the respective correction factor provided in the Feritscope’s certificate. The Feritscope measurements have been performed in the mid-width of the section along its longitudinal direction so that, according to Ortwein (2015), when the deformed width of the sample is smaller than 4 mm, the correction has to be applied twice since the mid-width measurement is affected by the two section’s edges. Taking into account that the deformed gauge-length sections present a width range between 3.25 mm and 4.42 mm for the different strain levels for which the Feritscope analysis is performed, the overall correction factor varies between 1.03 and 1.62. Moreover, the bi-linear calibration curve proposed by Ortwein (2015) is subsequently applied to transform the device measurements into α' -martensite content. Eight measurements have been performed on each deformed gauge-length section of the as-tested tensile samples (four measurements per section’s side) in order to obtain a representative value of the α' -martensite content. From the eight measurements the average value has been calculated (the maximum deviation from the average is 6.4% and it corresponds to the samples tested at 4 K).

- **Electron backscatter diffraction**

Electron backscatter diffraction (EBSD) can identify and map the phases present in the microstructure based on the diffraction of electrons, generated by the scanning electron microscope (SEM), from the specific crystallographic planes. It allows the visualization of the phases’ morphology and the calculation of their respective fraction on the mapped surface. The specimens have been extracted from the gauge length’s section of the deformed tensile-test samples. For each section, two cuts perpendicular to the sample’s longitudinal direction have been performed to obtain a single 11 mm-long specimen. The surface of the specimens has been prepared by mechanical grinding down to 1200 grit on SiC abrasive grinding papers and mechanical polishing in three steps down to 1 μm diamond solution, followed by colloidal silica polishing. For reasons of comparability with the Feritscope measurements, the prepared surface corresponds to one of the longitudinal-width planes

of the gauge-length section. In the present study, the EBSD analyses have been performed using a ZEISS Sigma Field Emission Gun Scanning Electron Microscope (FEG-SEM) with a detector of backscattered electrons. The acquired data have been evaluated with the Oxford Instruments AZTEC-EBSD software. The SEM has been operated at 20 kV using an aperture size of 240 μm and 16.5 mm of working distance with the specimen tilted by 70° . Each map corresponds to an area of approximately $455 \times 320 \mu\text{m}^2$ which has been acquired at a magnification of 250 and has been analysed using a step size of 0.75 μm . One analysis took approximately 10 hours with the before mentioned settings. The analyses reached a pixel indexing between 83.5% and 99.8%. Note that two databases have been used for the analysis: iron body-centred cubic for martensite, and iron face-centred cubic for austenite. The reason for using body-centred cubic for identifying the martensite is due to the fact that the c/a-ratio of its actual body-centred tetragonal lattice is very close to one (Naraghi, 2009) and the EBSD is not very sensitive to small variations of the crystallographic lattice. The selected batch of analysed specimens consists on 14 specimens which include for each material two deformation levels per tested temperature and a reference specimen in the undeformed state. Moreover, one single analysis has been performed for each specimen, and therefore, each result is based on a sample area of approximately 0.15 mm^2 . The volume fraction of martensite is obtained assuming that it is equal to its area fraction.

- **Quantitative light optical micrography**

Light optical microscopy allows to distinguish between phases with different crystallographic structure based on their different light reflection from the as-polished specimen's surface. Quantification analysis determines the fractions of the phases on the micrographs based on their different colour contrast. In the present study, the micrographs have been examined with a Carl ZEISS Optical Microscope connected to the ZEISS AxioCam MRc5 High Resolution Camera. Each micrograph covered an area of about $180 \times 135 \mu\text{m}^2$ which has been acquired at a magnification of 500, resulting in a pixel size of 0.13 μm . The light optical micrographs show the martensite in dark and austenite in light (see Fig. 19 in Section 6). The correspondence between the two colour-contrasts and the two phases has been obtained by comparing EBSD maps with light optical micrographs covering the same sample area (the comparison is not shown here for the sake of brevity). For the phase quantification an automatic image analysis using MATLAB software (MathWorks Inc., MA, United States) has been applied on the acquired micrographs. The analysis determines the martensite fraction from binary images which are obtained by image thresholding the processed micrographs. The code used for the determination of the content of martensite is included as *Supplementary Material*. The specimens used for the quantitative light optical microscopy analysis are the same analysed by EBSD. For this analysis, 24 micrographs per specimen have been acquired. For the sake of reproducibility, each of the 24 micrographs has been taken at approximately the same surface's position on the different specimens. From the 24 analyses per specimen, the martensite content for each specimen is based on an a total sample area of approximately 0.50 mm^2 . The volume fraction of martensite is obtained assuming that it is equal to its area fraction.

4 Flow behaviour: results

The uniaxial tension stress-strain curves, $\sigma - \varepsilon$, obtained from AISI 304L and AISI 316LN samples tested at different temperatures, $T = 4 \text{ K}$, 77 K and 300 K , are shown in Fig. 4. The stress is the Cauchy stress computed from the machine's load cell and the strain, hereinafter also referred to as raw strain, is the Hencky strain calculated from the displacement values acquired by the extensometer, as mentioned in Section 3.1. The imposed initial strain rate in the experiments is $\dot{\varepsilon} \approx 10^{-4} \text{ s}^{-1}$. The results correspond to uninterrupted tests performed with the Single-Section Sample (see Section 3.1). For the sake of clarity in the presentation of data we

only show one stress-strain curve per material and testing temperature. In this regard, note that the experiments showed good repeatability and the differences in the stress-strain characteristics obtained from different samples tested at the same temperature are small, and do not affect the discussion of results presented below. The comparison of the stress-strain curves obtained in this work with data available in the literature is performed in A. Moreover, recall that here we are studying the influence of temperature on the material mechanical behaviour, and all the samples for each material grade have been produced using the same manufacturing process (see Section 2). The reader is referred to the recent paper of Kvackaj et al. (2020) to obtain insights into the influence of rolling temperature, ambient or cryogenic, on the mechanical properties of AISI 316LN.

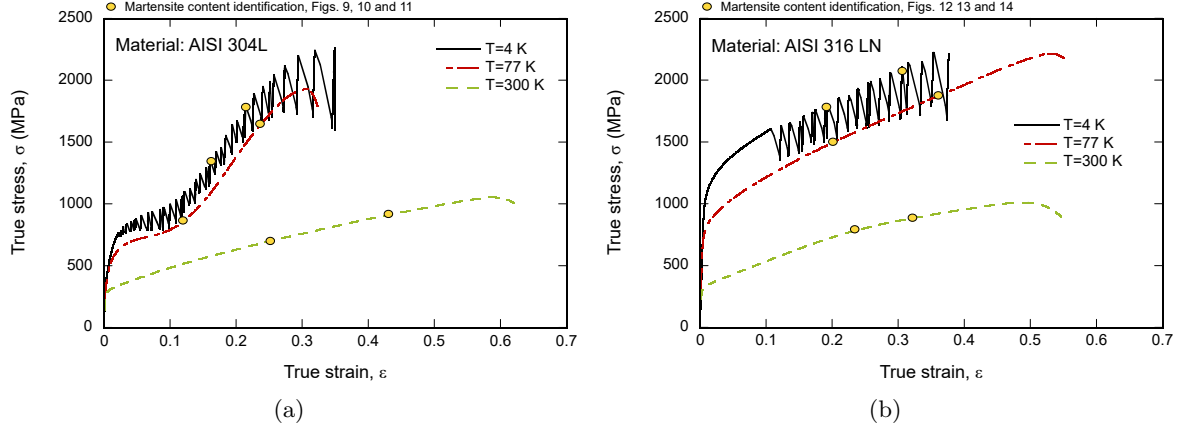


Figure 4: Stress-strain curves for three different testing temperatures $T = 4$ K, 77 K and 300 K. The imposed initial strain rate in the experiments is $\dot{\epsilon} \approx 10^{-4} \text{ s}^{-1}$. Results corresponding to: (a) AISI 304L and (b) AISI 316LN. The yellow markers indicate the values of strain corresponding to the EBSD maps of Figs. 7-12.

Fig. 4(a) shows the results corresponding to AISI 304L. At room temperature, the material displays an initial 0.2% offset yield strength of $\sigma_{YS} \approx 300$ MPa and quasi-linear strain-hardening, so that the necking forms at a strain of ≈ 0.51 and the ultimate tensile strength is $\sigma_{TS} \approx 1000$ MPa (both initial yield stress and ultimate tensile strength are determined following the procedure detailed in ASTM-E8/E8M-15a (2015) converting the values to true stress, necking is assumed to start when the ultimate tensile strength is reached). At 77 K the initial 0.2% offset yield strength is ≈ 350 MPa and the stress-strain characteristic displays a sigmoidal shape, with a sudden increase of the strain-hardening rate at a strain of ≈ 0.07 . The ultimate tensile strength is ≈ 1900 MPa and the necking strain ≈ 0.30 . The change of curvature of the stress-strain curve and loss of ductility are typical of a material in which the α' -martensite fraction increases rapidly with strain (Morris Jr et al., 1992; Skoczeń, 2001), as it will be shown later (see Fig. 13). At 4 K the stress-strain curve is further shifted upwards, and the plastic flow becomes discontinuous. The transition between continuous and discontinuous plastic flow is observed mainly in face-centred cubic metals and alloys at extremely low temperatures close to absolute zero. It is related to a change in the predominant type of moving dislocation from screw to edge character since the latter needs less thermal energy for its generation and motion. The phenomenon of serrated yielding occurs at sufficiently low strain rates, so the released heat during deformation can be easily removed by the cryogenic coolant and the motion of dislocation maintains its predominant edge character (Seeger, 1957; Obst and Nyilas, 1991). According to Obst and Nyilas (1991), this phenomenon has a mechanical nature: it is caused by the local catastrophic failure of barriers formed in the weakly excited lattice, whose collapse leads to a massive motion of dislocations. The failure occurs when the edge dislocations pile-ups give rise to stress concentrations which are high enough to overcome the cohesive strength. The massive motion of dislocations occurs locally at the unblocked lattice barriers, then the dislocations move until they reach the next obstacle and the whole process is repeated. Macroscopically, this

process leads to simultaneous stepwise increases of strain and abrupt drops of stress, the former consisting in slip bands travelling along the sample (Tabin et al., 2016, 2019). In the course of strain hardening the local breakdown of lattice barriers becomes more difficult (Obst and Nyilas, 1991), thus more pronounced serrations –greater strain increment between individual serrations and higher load drop– are observed in Fig. 4 for higher strains.

Fig. 4(b) shows the results corresponding to AISI 316LN. At room temperature, the stress-strain curve is similar to AISI 304L, with quasi-linear strain-hardening and significant ductility, the necking strain and the ultimate tensile strength being ≈ 0.32 and ≈ 900 MPa, respectively. At 77 K the flow stress of AISI 316LN rises significantly –the initial 0.2% offset yield strength and the ultimate tensile strength reaching ≈ 700 MPa and ≈ 2100 MPa, respectively– while keeping linear strain hardening and large ductility (unlike AISI 304L). Decreasing the testing temperature up to 4 K further increases the material flow stress and decreases the necking strain so that the ultimate tensile strength corresponds to a strain of ≈ 0.34 . Notice that the flow stress shows serrations for strains greater than 0.1. Nevertheless, for some of the experiments performed with the AISI 316LN samples at 4 K, the serrated yielding started earlier, for $\varepsilon \approx 0.05$.

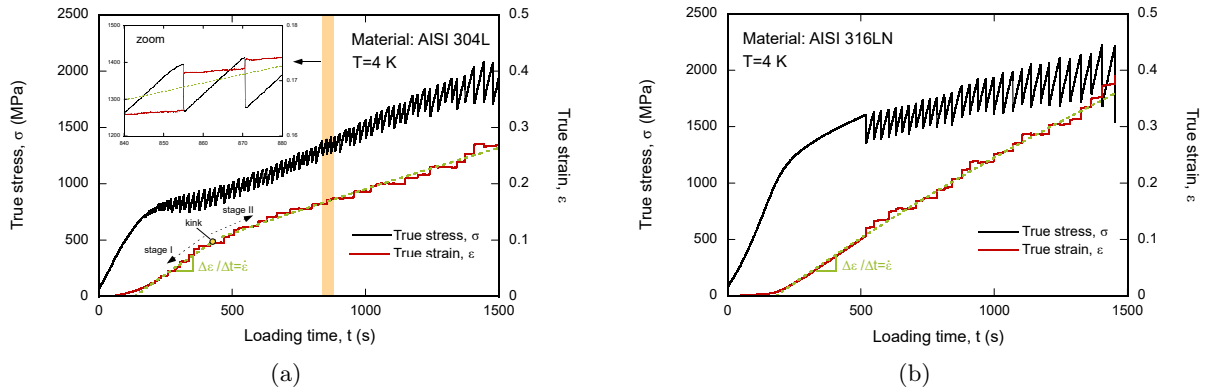


Figure 5: Temporal evolution of the stress and the strain for experiments performed at 4 K using the Single-Section Sample. The green dashed lines represent the uniform strain on the sample’s gauge-length section during plastic deformation (two-stage strain rate for the case of AISI 304L within the range of strains analysed in the figure). Results corresponding to: (a) AISI 304L and (b) AISI 316LN.

As already discussed, the discontinuous plastic flow leads to simultaneous stepwise increases of strain and abrupt drops of stress with respect to time during the tests, so that the strain at 4 K is not uniform along the sample’s gauge-length but rather has a local nature, as illustrated in Fig. 5 which shows the evolution of the stress (black solid line) and the raw strain (red solid line) with the loading time for the same tests at 4 K presented in Fig. 4. As mentioned before, the raw strain is calculated from the displacement values obtained with the extensometer. The graphs also picture the evolution of the uniform strain which is computed averaging the strain rate over time along the sample’s gauge length (green dashed line), where the strain rate is calculated as the ratio between the increase of strain within a time interval. Notice that both AISI 304L and AISI 316LN samples show the same uniform strain rate at the beginning of loading (recall that the initial strain rate is $\dot{\varepsilon} \approx 10^{-4} \text{ s}^{-1}$). However, while for AISI 316LN samples the strain rate is *roughly* constant during the whole loading process –actually, there is a gradual decay of the strain rate due to the sample elongation–, in the case of AISI 304L samples tested at cryogenic temperatures the uniform strain rate suddenly decreases to half of its value at $\varepsilon \approx 0.07$ (at the kink indicated in Fig. 5(a)). Notice that this is the strain value for which the stress-strain curve changes from concave-downwards to concave-upwards, see Fig. 4(a). The drop of the strain rate occurs because the sections of the sample with $\varepsilon > 0.07$ need higher stress increment to keep deforming than the sections with a lower strain level, so that the contribution of the sections of the sample beyond the gauge length to the total sample elongation increases. Consequently, since the velocity of the machine’s cross-head is constant, the uniform strain rate on the sample’s

gauge length decreases.

Fig. 6 shows experimental results for AISI 304L obtained at 77 K with the Double-Section Sample. Notice that the stress-strain curves computed from interrupted tests performed with the Double-Section Sample –coloured lines– and from uninterrupted tests carried out with the Single-Section Sample –black lines– are very similar. Solid coloured lines correspond to the results of the 4 mm width gauge-length section of the sample, and dashed coloured lines to the 4.5 mm width section. Each colour corresponds to the results obtained from a given Double-Section Sample. As anticipated in Section 3.1, performing four interrupted tests enables to obtain a set of eight distributed strain levels to measure the α' -martensite content in the samples before the ultimate tensile strength of the material is attained: two levels of strain at the end of each test, corresponding to the two gauge-length sections of the sample. Similar observations are derived from the tests performed at 4 K and 300 K, and also from the interrupted experiments conducted with the Double-Section Sample designed for AISI 316LN.

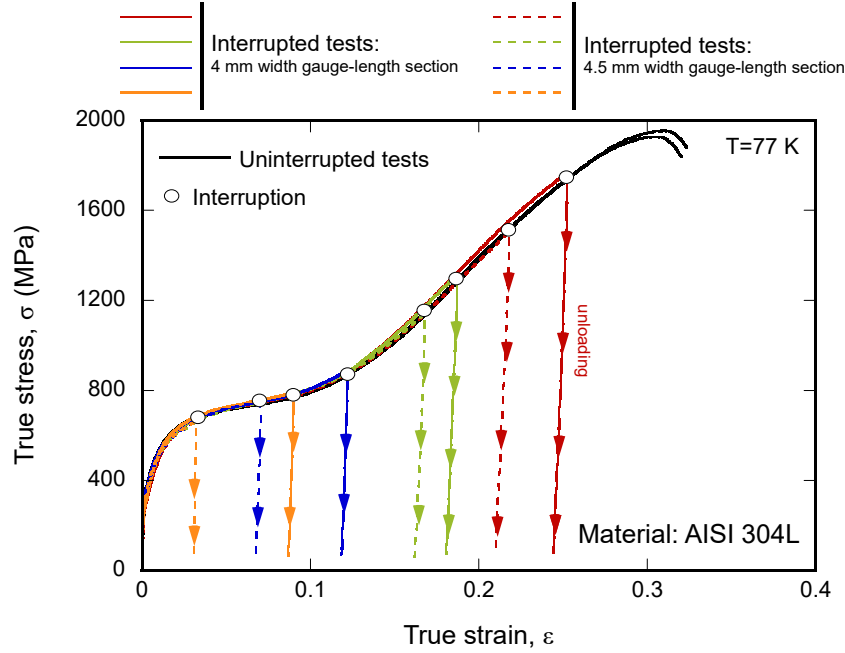


Figure 6: Experimental results for AISI 304L at 77 K. Comparison between stress-strain curves obtained from interrupted tests performed with the Double-Section Sample –coloured lines– and from uninterrupted tests carried out with the Single-Section Sample –black lines–. Solid coloured lines correspond to the results of the 4 mm width gauge-length section, and dashed coloured lines correspond to the 4.5 mm width section. Each colour corresponds to the results obtained from a given Double-Section Sample.

The EBSD maps obtained from Double-Section Samples, which were used in interrupted experiments performed at different testing temperatures, are shown in Figs. 7-12. The results for AISI 304L are pictured in Figs. 7-9, and the results for AISI 316LN in Figs. 10-12, respectively. The colour coding of the EBSD maps is such that the martensite is in blue and the austenite is in red. The observed morphology of the α' -martensite phase consists of the low-carbon lath-type martensite. The laths tend to align parallel to one another forming a packet arrangement. Moreover, several lath-packages with different orientations normally coexist in a single austenite grain (Vander Voort et al., 2004; Naraghi, 2009). From the EBSD maps analysed here, the lath-packages are distinguishable for specimens containing relative small contents of martensite (up to approximately 30%). Specimens containing a high content of martensite (higher than 80%) show austenite grains which are quasi-fully transformed into martensite. It can be observed that the α' -martensite phase tends to form at or nearby stacking faults, which are recognized as parallel black lines inside the austenite grains (see Fig. 10(b) where the quasi-monochromatic

red map allows an easy identification of the stacking faults). The density of stacking faults is noted to be increased with increasing strain. Moreover, the EBSD maps corresponding to high level of deformations show grains greatly deformed with a tendency to elongate along the rolling direction (Figs. 7(b)-12(b)).

Figs. 7(a)-(b) show images for an AISI 304L sample tested at 300 K corresponding to two values of strain, 0.25 and 0.43, respectively. While the amount of martensite is not large, these images make apparent the increasing formation of martensite in AISI 304L due to the accumulation of plastic deformation at room temperature. On the other hand, the amount of martensite significantly increases at cryogenic temperatures. Figs. 8(a)-(b) show EBSD maps corresponding to a sample tested at 77 K, which reveal a large martensite content for a relatively low strain of ≈ 0.12 , and quasi-complete transformation of the austenite grains into martensite at a strain of ≈ 0.24 . Moreover, the results obtained from the tests performed at 4 K, see Figs. 9(a)-(b), are similar to the EBSD maps of the tests at 77 K with the material becoming almost fully martensitic for strains greater than 0.2.

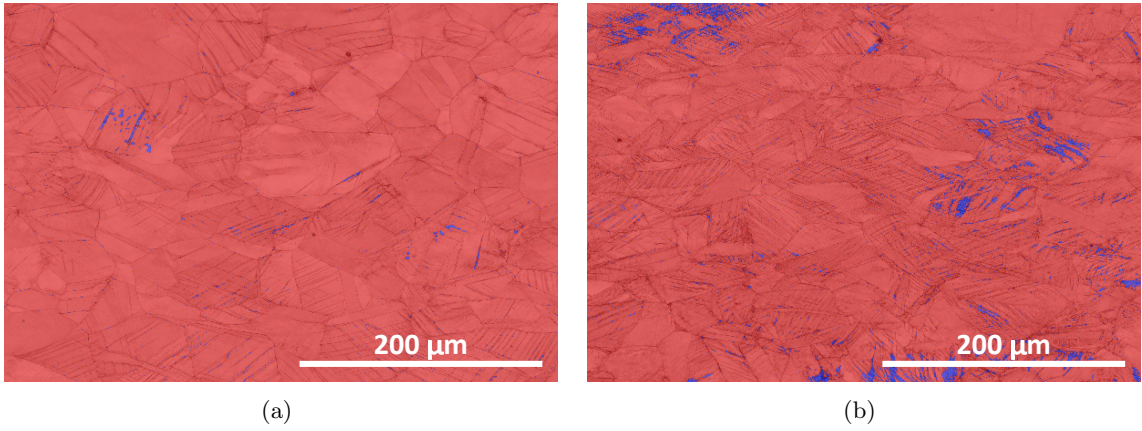


Figure 7: EBSD maps of deformed AISI 304L Double-Section Sample tested at 300 K. Rolling direction is horizontal in the micrographs. Colour coding: martensite in blue and austenite in red. The maps correspond to different values of strain indicated in Fig. 4(a) with yellow markers: (a) $\varepsilon \approx 0.25$ and (b) $\varepsilon \approx 0.43$.

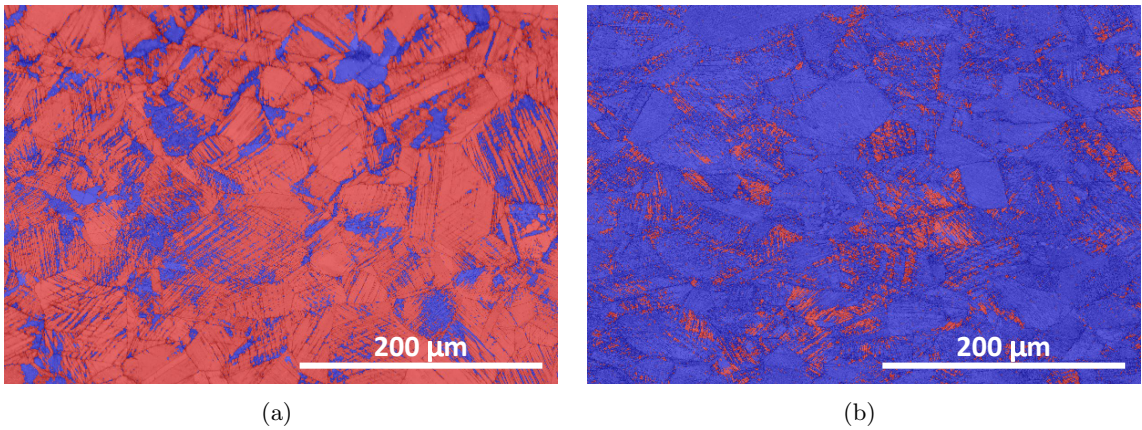


Figure 8: EBSD maps of deformed AISI 304L Double-Section Sample tested at 77 K. Rolling direction is horizontal in the micrographs. Colour coding: martensite in blue and austenite in red. The maps correspond to different values of strain indicated in Fig. 4(a) with yellow markers: (a) $\varepsilon \approx 0.12$ and (b) $\varepsilon \approx 0.24$.

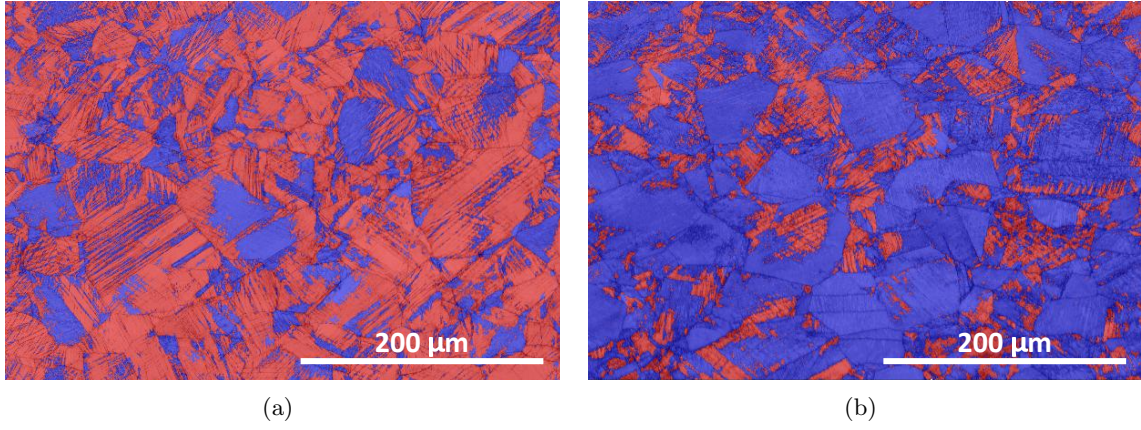


Figure 9: EBSD maps of deformed AISI 304L Double-Section Sample tested at 4 K. Rolling direction is horizontal in the micrographs. Colour coding: martensite in blue and austenite in red. The maps correspond to different values of strain indicated in Fig. 4(a) with yellow markers: (a) $\varepsilon \approx 0.16$ and (b) $\varepsilon \approx 0.22$.

Figs. 10(a)-(b) shows EBSD maps for an AISI 316LN sample tested at 300 K which reveal that, unlike in the case of AISI 304L, negligible amount of martensite is formed in this steel grade at room temperature. On the other hand, decreasing the testing temperature promotes the martensitic transformation, yet, the content of martensite in AISI 316LN samples tested at cryogenic temperatures is significantly lower than in the case of AISI 304L. Namely, the results presented in Figs. 11(a)-(b) for a test carried out at 77 K show that for a strain of ≈ 0.20 the martensite content is virtually negligible, and that increasing the material straining up to 0.36 only causes a modest portion of austenite to transform into martensite. Moreover, the EBSD maps of Figs. 12(a)-(b) show that the formation of martensite at 4 K starts at a lower level of strain in comparison with the test performed at 77 K (compare Figs. 11(a) and 12(a)), and the transformation seems to be faster (compare Figs. 11(b) and 12(b)).

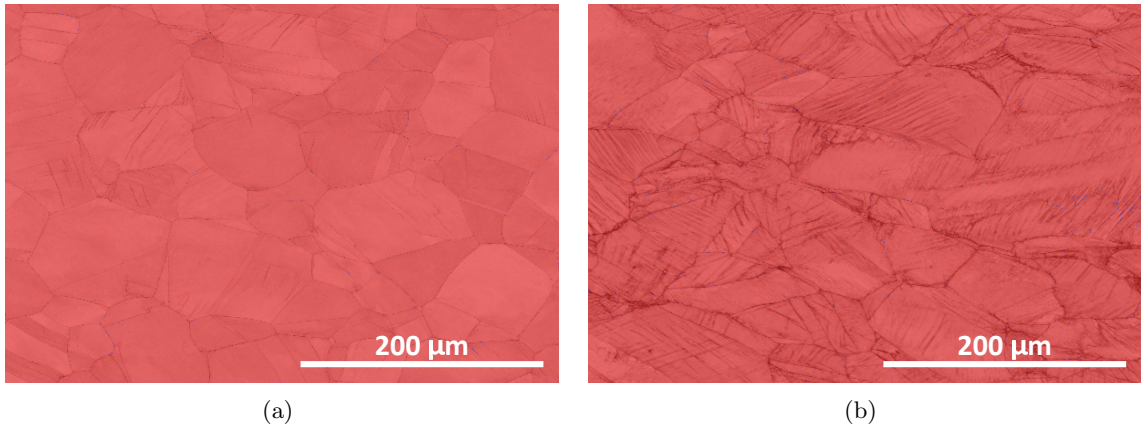


Figure 10: EBSD maps of deformed AISI 316LN Double-Section Sample tested at 300 K. Rolling direction is horizontal in the micrographs. Colour coding: martensite in blue and austenite in red. The maps correspond to different values of strain indicated in Fig. 4(b) with yellow markers: (a) $\varepsilon \approx 0.23$ and (b) $\varepsilon \approx 0.32$.

Quantitative measurements for the evolution of the martensitic transformation with the strain (elastic strains are neglected, so that all the strain is considered to be plastic) are shown in Figs. 13 and 14. The data correspond to measurements obtained with all the three methods: magnetic induction, electron backscatter diffraction and quantitative light optical micrography (black, red and green markers, respectively). Notice that the results obtained with the three

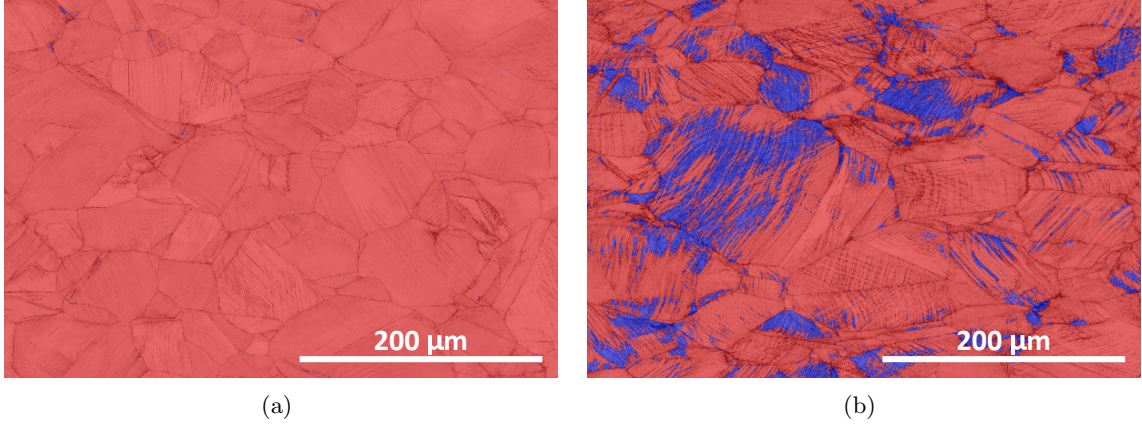


Figure 11: EBSD maps of deformed AISI 316LN Double-Section Sample tested at 77 K. Rolling direction is horizontal in the micrographs. Colour coding: martensite in blue and austenite in red. The maps correspond to different values of strain indicated in Fig. 4(b) with yellow markers: (a) $\varepsilon \approx 0.20$ and (b) $\varepsilon \approx 0.36$.

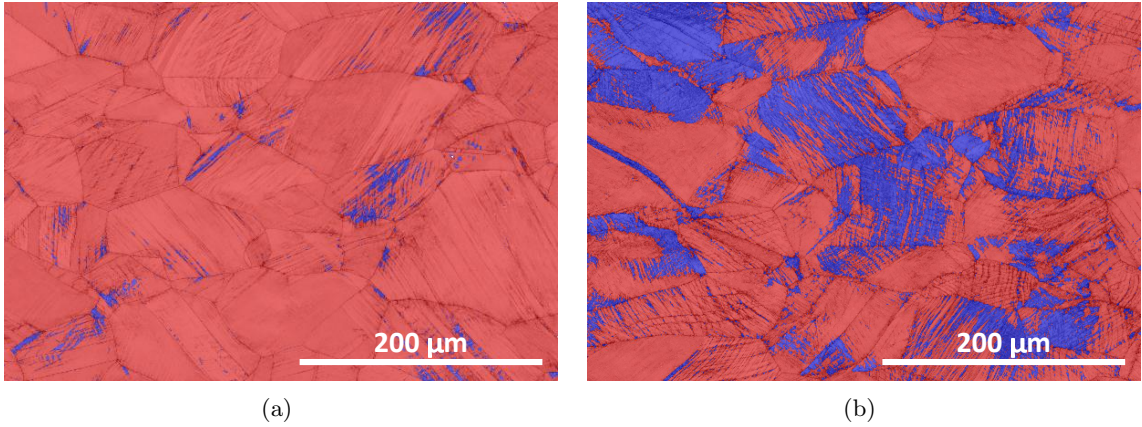


Figure 12: EBSD maps of deformed AISI 316LN Double-Section Sample tested at 4 K. Rolling direction is horizontal in the micrographs. Colour coding: martensite in blue and austenite in red. The maps correspond to different values of strain indicated in Fig. 4(b) with yellow markers: (a) $\varepsilon \approx 0.18$ and (b) $\varepsilon \approx 0.31$.

methods are similar, for the three testing temperatures (300 K, 77 K and 4 K) and both materials, which shows the robustness and reliability of the measurements. The comparison of the martensite content obtained in this work with data available in the literature is performed in A.

Fig. 13 pictures the results corresponding to AISI 304L. At room temperature the martensitic transformation starts at $\varepsilon \approx 0.05$ and increases with the strain so that for $\varepsilon \approx 0.4$ the volume fraction of martensite $f^{\alpha'}$ reaches $\approx 5\%$. The evolution of the martensite content with the strain is very similar for 77 K and 4 K, with the martensite volume fraction $f^{\alpha'}$ reaching $\approx 70\%$ for a strain of ≈ 0.2 (see Figs. 8(b) and 9(b)). The rapid martensitic transformation displayed by AISI 304L at cryogenic temperatures explains the sigmoidal shape of the stress-strain curves shown in Fig. 4, so that the higher strength of the martensitic phase, in comparison with the austenite, is responsible for the sudden increase of the strain-hardening rate of the material. To the authors knowledge these are the first measurements available in the open literature for the martensite content in 304L tensile samples tested at 4 K. To the authors' knowledge it is also the first time the evolution of the martensite content with the strain shows almost an equal behavior when reducing the temperature, as it is the case of the results obtained in this work for AISI 304L at 77 K and 4 K. This fact seems to indicate that for this grade the transformation process is already saturated at 77 K, so the formation of martensite cannot occur either earlier

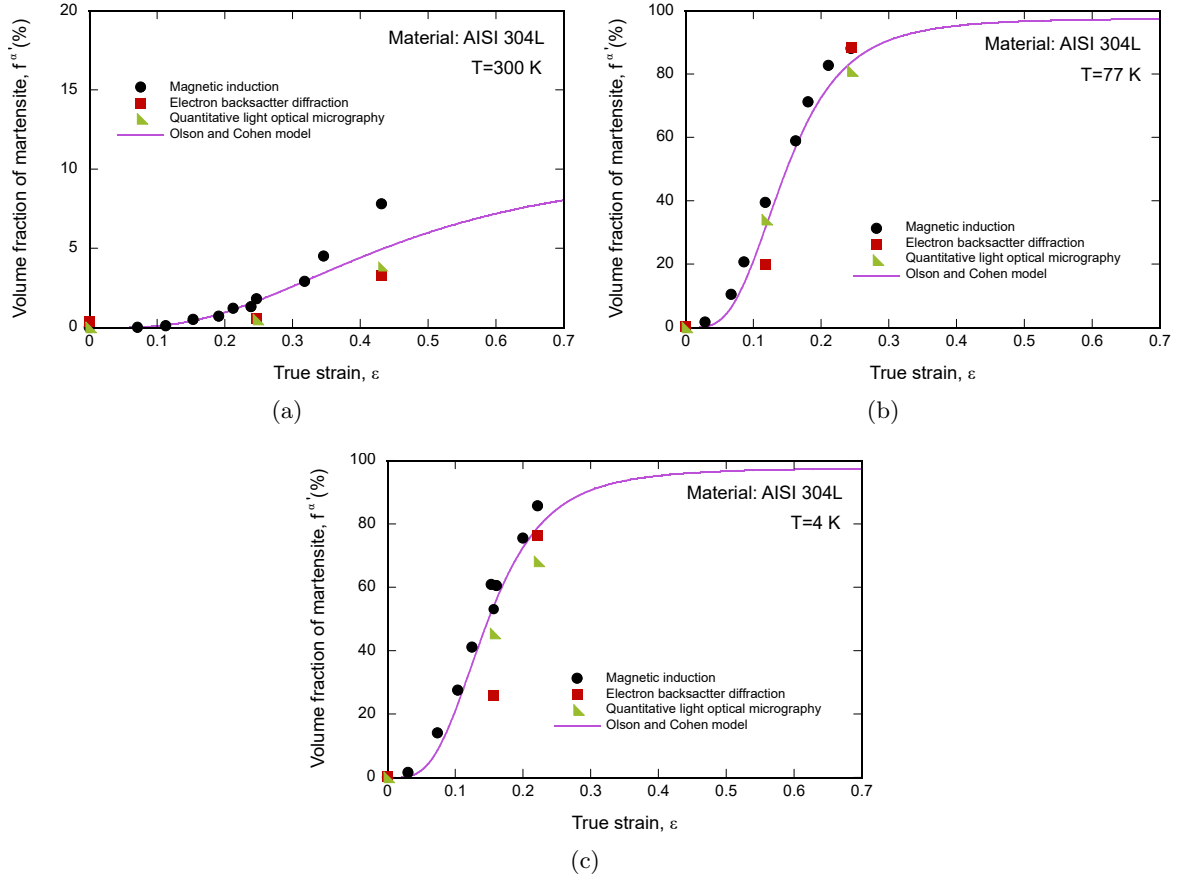


Figure 13: Evolution of the volume fraction of martensite $f^{\alpha'}$ with the strain ϵ in AISI 304L samples tested at different temperatures: (a) 300 K, (b) 77 K and (c) 4 K. The experimental data correspond to measurements obtained with all the three methods: magnetic induction, electron backscatter diffraction and quantitative light optical micrography (black, red and green markers, respectively). The solid line corresponds to the kinetic relation of Olson and Cohen (1975) which has been fitted to the experimental data (see Tables 2 and 3).

or develop faster at 4 K.

Fig. 14 presents the results for AISI 316LN. The three measurement methods detected martensite formation at room temperature lower than 0.3% (which is within the error of the experimental measure), which evidences the higher stability of this material, in comparison with AISI 304L, against martensitic transformation (see Fig. 10 versus Fig. 7). Moreover, unlike in the case of AISI 304L, there are significant differences in the kinetics of the martensitic transformation at 77 K and 4 K, so that the formation of martensite starts earlier and develops faster at the lower temperature. Namely, at 77 K the volume fraction of martensite for $\epsilon = 0.2$ and 0.3 is $\approx 2\%$ and 15%, while at 4 K is $\approx 7\%$ and 25%, respectively. Nevertheless, the rate of the transformation and the volume fraction of austenite transformed into martensite are smaller than in the case of AISI 304L. In this regard, Morris Jr et al. (1992) and Skoczni (2001) stated that a slow martensitic transformation does not produce a significant change on the strain-hardening rate of the material and enhances tensile ductility, in agreement with the stress-strain curves shown in Fig. 4(b) –compare the results with Fig. 4(a) for AISI 304L–. To the authors knowledge these are the only measurements available in the literature for the martensite formation in 316LN samples tested at 4 K.

It is noticeable that, for both grades, the maximum deviation from the average content between the eight measurements performed using the magnetic induction method has been obtained in the case of samples tested at 4 K (see item **Magnetic induction** in Section 3.2). It indicates that the local nature of the strain at 4 K, as already discussed in a previous paragraph,

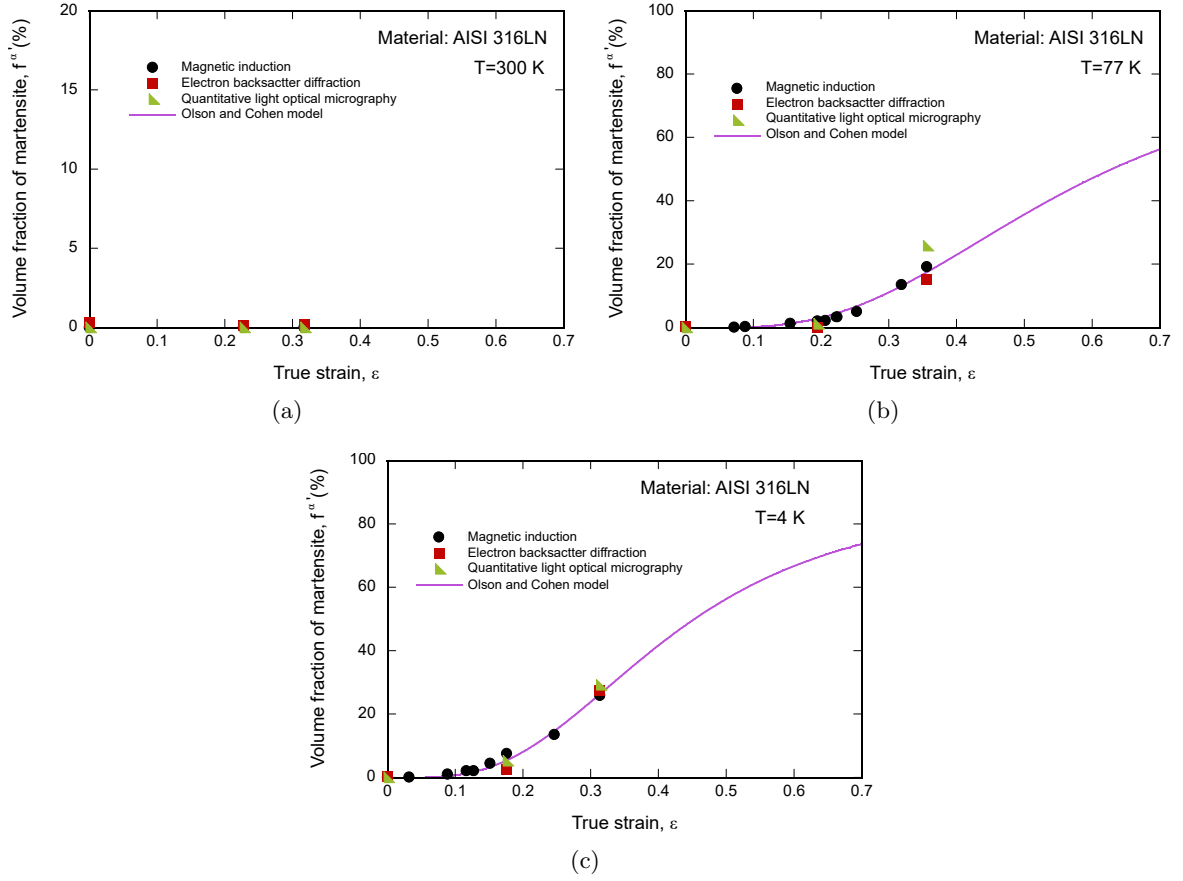


Figure 14: Evolution of the volume fraction of martensite $f^{\alpha'}$ with the strain ϵ in AISI 316LN samples tested at different temperatures: (a) 300 K, (b) 77 K and (c) 4 K. The experimental data correspond to measurements obtained with all the three methods: magnetic induction, electron backscatter diffraction and quantitative light optical micrography (black, red and green markers, respectively). The solid line corresponds to the kinetic relation of Olson and Cohen (1975) which has been fitted to the experimental data (see Tables 2 and 3).

leads to a less homogeneous distribution of martensite along the sample's gauge-length section in comparison with the samples tested at 77 K and 300 K where the plastic flow is continuous (see Fig. 4).

The experimental data for the evolution of the martensite volume fraction with the strain (recall that elastic strains are neglected) are fitted using the sigmoidal-type kinetic relationship proposed by Olson and Cohen (1975), see Figs. 13 and 14. Olson and Cohen model is given by equation (1), where the parameter ζ represents the rate of shear-band formation and ξ indicates the probability that an intersection of shear bands will result in the nucleation of martensite.

$$f^{\alpha'} = 1 - e^{-\xi(1 - e^{(-\zeta\epsilon)})^{4.5}} \quad (1)$$

The values of the model parameters are given in Tables 2 and 3. The identification process has been carried out by assuming a temperature-evolution function for each parameter. The selected functions are based on the former experimental data provided by Olson and Cohen (1975), who showed a dependence of the parameters ζ and ξ on temperature –see Fig. 2 therein– which in this work are approximated with a half sigmoid and a full sigmoid function, respectively, see equation (2).

$$\zeta = \frac{\zeta_2}{1 + e^{[-\zeta_3(T - \zeta_1)]}} - \frac{\zeta_2}{2}; \quad \xi = \frac{\xi_2}{1 + e^{[-\xi_3(T - \xi_1)]}}; \quad \zeta_1 \geq 0; \quad \zeta_2 \geq 0; \quad \zeta_3 \leq 0; \quad \xi_2 \geq 0; \quad \xi_3 \leq 0 \quad (2)$$

	AISI 304L			AISI 316LN		
	T = 300 K	T = 77 K	T = 4 K	T = 300 K	T = 77 K	T = 4 K
ζ	4.49	7.81	7.81	0.37	2.64	3.38
ξ	0.10	3.74	3.74	0.00	1.80	2.08

Table 2: Identification of the parameters corresponding to the kinetic relation of Olson and Cohen (1975), see equation (1), used in the fitted curves shown in Fig. 13 and Fig. 14.

	AISI 304L	AISI 316LN
ζ_1	$3.36 \cdot 10^2$	$3.36 \cdot 10^2$
ζ_2	$1.56 \cdot 10^1$	$9.32 \cdot 10^2$
ζ_3	$-3.66 \cdot 10^{-2}$	$-4.36 \cdot 10^{-5}$
ξ_1	$2.71 \cdot 10^2$	$1.24 \cdot 10^2$
ξ_2	3.74	2.10
ξ_3	$-1.25 \cdot 10^{-1}$	$-3.73 \cdot 10^{-2}$

Table 3: Identification of the parameters corresponding to the kinetic relation of Olson and Cohen (1975), see equation (2), used in the fitted curves shown in Fig. 13 and Fig. 14.

5 Fracture behaviour: experimental techniques

5.1 Mechanical tests

Elastic-plastic fracture toughness tests with fatigue-precracked Compact Samples for the opening mode of loading (i.e. Mode I) have been carried out at liquid nitrogen temperature ($T = 77$ K) and at liquid helium temperature ($T = 4$ K), according to the standard ASTM-E1820-20a (2020). All the fatigue pre-cracking and fracture toughness tests have been performed at CERN. The tests have been used to compute crack-growth resistance $J - R$ curves following the so-called “resistance curve procedure” (see ASTM-E1820-20a (2020)), i.e. using a single Compact Sample to obtain the complete $J - R$ curve. Experiments at room temperature have not been performed since, for the two analysed materials, samples with thickness greater than 12 mm, that is the thickness of the plates from which all samples tested in this work are extracted (see Section 2)), are needed to obtain fracture toughness values that are independent of the sample size (the threshold thickness for AISI 304L is estimated to be around 25.4 mm based on the work of Sa et al. (2005)). Additionally, since here we are only considering fracture toughness of Mode I, the reader is referred to the recent paper of Wang et al. (2020) to obtain insights into the fracture behaviour of metals under mixed mode loading.

The same servo-hydraulic testing machine and cryostat used for the uniaxial tensile tests (Section 3.1) have been employed to perform the fatigue pre-cracking and the fracture toughness experiments. The basic design lines of the tension testing clevis were presented by Malik (2021). Moreover, the design of the Compact Sample consists of an “alternative specimen” according to ASTM-E1820-20a (2020), with a width-to-thickness ratio of $W/B = 4$ ($W = 40$ mm and $B = 10$ mm, see the corresponding dimensions in Fig. 15). The Compact Samples have been extracted from the mid-thickness of each material plate with the load-line axis parallel to the rolling direction (L-T direction according to the standard ASTM-E1823 (2020)). They have been machined using spark-erosion for the in-plane geometry and subsequent milling to obtain the pin holes and the desired thickness.

Fatigue pre-cracking of the Compact Samples has been carried out at room temperature according to the standard ASTM-E1820-20a (2020), following the same procedure for both

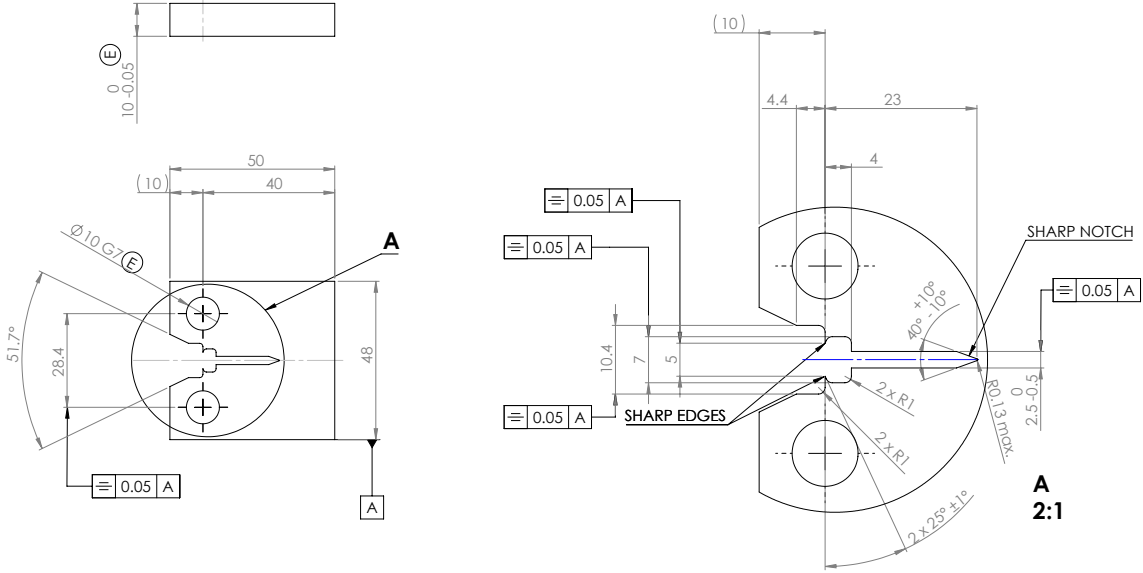


Figure 15: Design of the Compact Sample used in the fracture toughness tests.

materials, with the purpose of obtaining a sharp precrack. Monitoring of the precrack has been performed with a portable optical microscope (Wi-Fi Digital Microscope RS PRO 913-2478). The precrack has been produced by cyclic loading the sample with a sinusoidal waveform at a maximum frequency of 30 Hz under force control using a load ratio of $R = 0.1$. The first step of the pre-cracking has been conducted using a maximum load of 1 kN. The transition to the second step has been done when the number of cycles is $\approx 10^5$. For the second step a maximum load of 4 kN has been selected. The test was stopped at the moment in which the precrack reached approximately 3 mm (from the sharp notch, see Fig. 15), which corresponds to an initial crack size of $a_0 \approx 0.65W \approx 26$ mm (distance from load-line to fatigue precrack tip, see Fig. 15). Hence, the remaining ligament is $b_0 \approx W - a_0 \approx 14$ mm. Next, side grooves with a total thickness reduction of approximately 2 mm were machined. The machining has been performed using spark-erosion since, according to Weiss and Nyilas (2006), it leads to a straighter crack front during the fracture toughness test.

The elastic-plastic fracture toughness tests have been performed according to the standard ASTM-E1820-20a (2020), following the “resistance curve procedure” (as mentioned before). The load was applied under displacement control of the machine’s cross head at a speed of 0.5 mm/min, and it was measured by the machine’s load cell. A number of unloading-reloading sequences have been performed along the test every 0.14 mm of the machine’s cross-head displacement, with a force reduction range of 2 kN, see Fig. 16. A cryogenic clip gage (Epsilon 3541-005M-040M-LT) has been used to measure the load-line displacement. While the load and load-line displacement values have been continuously measured during the tests, the crack size and J values have been calculated a posteriori when computing the crack-growth resistance $J - R$ curves (as discussed in a later paragraph). As mentioned before the cryogenic test setup described in Section 3.1 has been employed for all the tests. Three fracture toughness tests per material and testing temperature have been conducted. The tests are stopped shortly after the point of maximum force is exceeded, which is an indication that the crack has propagated, and before the clip gage reaches the maximum measuring range of 5 mm. The final crack size after testing has been marked by heat tinting the sample at 400°C for 1 hour. Subsequently, the sample has been fractured in order to analyse the crack. Identification of the initial and final crack sizes has been conducted using an optical microscope. The initial crack size corresponds to the end of the flat fatigue precracked area, while the final crack size is marked by the colouration from heat tinting (see Fig. 18).

The load and load-line displacement measurements obtained from the fracture toughness experiments have been used to compute the crack-growth resistance $J - R$ curves. From each

$J - R$ curve the fracture toughness parameter J_Q has been determined according to the standard ASTM-E1820-20a (2020). If all the qualification requirements of the standard are fulfilled a qualified value of J_Q is obtained, which represents the crack growth resistance near the onset of ductile and stable crack extension (at 0.2 mm of crack extension beyond initial crack tip blunting, see Fig. 17). This parameter qualified as size-independent plane-strain J_{Ic} (crack-extension resistance under conditions of crack-tip plane-strain) provided that the size criteria specified in Sections A9.10.1 and A9.10.2 of the standard ASTM-E1820-20a (2020) are also met. Moreover, Section A9.11 of the standard provides an equation which allows to obtain the stress intensity factor $K_{J_{Ic}}$ calculated from J_{Ic} : $K_{J_{Ic}} = \sqrt{(EJ_{Ic})/(1 - \nu^2)}$, where here the Young's modulus and Poisson's ratio are $E = 206$ GPa and $\nu = 0.3$, respectively. This formula makes possible an estimation of the crack-extension resistance near the onset of stable crack extension under dominant elastic conditions, which is very useful for predominantly-elastic applications when the sample tests do not yield valid K_{Ic} results (Joyce and Tregoning, 2000). To be noted the difference between $K_{J_{Ic}}$ and K_{Ic} , where the former is evaluated using J_{Ic} and the latter is directly measured (the reader is referred to the standard ASTM-E399 (2020) for a direct determination of K_{Ic}).

As mentioned before, four different methods have been used for the calculation of the $J - R$ curves, which is an original methodological contribution of this paper:

- **ASTM Compliance Method.** The *official* procedure provided by the standard ASTM-E1820-20a (2020) is an elastic-compliance approach based on the assumption that as the crack grows the sample becomes less stiff, leading to an increase of the unloading compliance with respect to the previous unloading. The crack length is related to the unloading compliance using the equations provided in the Appendix of the standard ASTM-E1820-20a (2020) devoted to the Compact Sample (Appendix 2 - Special requirements for testing compact specimens), which includes an adjustment procedure to improve the estimation of the initial crack size. The adjustment is based on a third-order polynomial regression fitting of crack size versus J values (Section A9.3 therein).
- **W-N Compliance Method.** This approach was developed in the works of Nyilas et al. (1998) and Weiss and Nyilas (2006), and includes two improvements with respect to the ASTM Compliance Method. On the one hand, it proposes a solution for the so-called apparent negative crack extension (a problem not addressed by the ASTM Compliance Method), which results from an experimentally-measured continuously increasing stiffness with respect to previous unloadings at the initial stage of the test (Voss, 1983; Rosenthal et al., 1990; Seok, 2000). While there is not a general consensus about the causes behind the apparent negative crack extension, Nyilas et al. (1998) and Weiss and Nyilas (2006) mainly attributed the initial increase of the sample's stiffness to the strain hardening of the plastic zone ahead of the precrack tip during blunting, which does not imply actual crack growth. Thus, the W-N Compliance Method identifies the crack initiation when the overall stiffness changes from an increasing to a decreasing behaviour, i.e. when the blunted precrack turns into ductile tearing. The data points before the designated crack initiation are not included in the $J - R$ curve, and therefore, no negative crack growth is predicted. On the other hand, the W-N Compliance Method improves the estimation of the crack lengths, replacing the experimentally-measured compliances by the so-called "corrected compliance function" of load-line displacement, which results from the construction of a second-order polynomial regression of stiffness versus displacement at the onset of unloading. The specific form of the polynomial regression is determined by imposing three conditions: (i) the displacement at the onset of the last unloading is associated with the compliance calculated with the equation (A2.11) of the standard ASTM-E1820-20a (2020) using the measured final crack length, (ii) the displacement at the crack initiation is associated with the compliance calculated with the equation (A2.11) of the standard ASTM-E1820-20a (2020) using the measured initial crack length, and (iii) the maximum of the polynomial regression corresponds with the displacement associated to the crack

initiation. Note that the measured initial crack length rather corresponds to the sharp precrack than to the blunted precrack. However, since the sharp precrack is readily measurable using optical microscopy, see Fig. 18, the “corrected compliance function” directly associates the sharp precrack with the crack initiation for the sake of simplicity. Generally, the difference between the blunted precrack and the sharp precrack is between $50\text{ }\mu\text{m}$ and $400\text{ }\mu\text{m}$ (Tarafder and Tarafder, 2006). Moreover, the W-N Compliance Method relates the values of J with the improved estimation of the crack extensions and the entire $J - R$ curve is shifted so that the crack initiation point coincides with the “construction line” (also referred to as blunting line). After the shifting, the computed value for the initial crack length is calculated as the crack length corresponding to a crack extension $\Delta a = 0$ (see Fig. 20), which is smaller than the experimental measurement of the initial crack length due to the simplification of associating the sharp precrack with the crack initiation point.

- **Modified W-N Compliance Method.** This approach is an original contribution of this paper, and the main difference with respect to the W-N Compliance Method is that the values of J are re-calculated using the improved estimation of the crack lengths obtained from the “corrected compliance function” of displacement. Moreover, the “corrected compliance function” associates the crack initiation with the blunted precrack, instead of with the sharp precrack, using an iterative procedure. The iterative procedure starts assuming that the blunted precrack is equal to the sharp precrack, the simplification used in the W-N Compliance Method. Once the $J - R$ curve is obtained, instead of performing the shifting to the blunting line, a new estimation of the blunted precrack is calculated as the sum of the measured initial crack length and the ratio between the J value of the current crack initiation point and the slope of the blunting line, indicated in Fig. 17(a). The iterative process is then re-started, so that in each iteration, “corrected compliance function”, crack lengths, J values, $J - R$ curve and blunted precrack are updated. The iterative process ends when the difference between current and previous estimation for the blunted precrack is lower than 10^{-5} mm , so that this iterative process ensures that the crack initiation point coincides with the blunting line and the computed initial crack length equals the measured initial crack length.
- **ASTM Normalization Method.** The Normalization technique enables direct estimation of the crack lengths from a test performed under continuous displacement increase, so that unloading-reloading sequences are not needed and estimations of intermediate crack lengths are based on the load versus load-line displacement record, as well as on the initial and final measured crack sizes (Herrera and Landes, 1990; Landes et al., 1991; Lee, 1995; Joyce, 2001). The Normalization method which has been used in this work is described in Annex 15 of the standard ASTM-E1820-20a (2020). Since the fracture toughness tests presented in this paper have been performed according to the “resistance curve procedure”, the data of the unloading-reloading sequences had to be removed. Specifically, we have retrieved for the analysis the whole record of load and displacement until the onset of the first unloading, one data-point between each unloading sequence, and the point corresponding to the onset of the last unloading, see Fig. 16.

The code used for construction of the crack-growth resistance $J - R$ curves, and the identification of the fracture toughness parameters J_Q and K_{JQ} , is included as *Supplementary Material*. The Potential Difference Method has not been used in this work due to the difficulty that it presents to accurately differentiate the potential drop signal caused by crack extension from the one caused by plasticity for a ductile crack growth (Zhu and Joyce, 2012; Park et al., 2019), as well as due to the high time- and resources-consumption that the calibration of this method implies at cryogenic temperatures (because it is necessary to relate potential drop with the crack size). Moreover, recall that all the fracture toughness tests are performed at low loading rate, so that the reader is referred to the recent papers of Pham and Iwamoto (2015, 2018) to obtain

insights into the influence of rate effects on the fracture behaviour of 304 austenitic stainless steel.

5.2 Martensite measurements

Light optical micrography inspections have been carried out to investigate the extent of the α' -martensitic transformation near the fracture surface of the Compact Samples after testing. The analysis encompasses samples of both materials, AISI 30L and AISI 316LN, tested at 77 K and 4 K. The samples selected for the analysis have been cut into two parts along their mid-thickness plane. One cutting plane for each specimen has been metallographically prepared. The cut and metallographic preparation have been carried out at CERN following the same procedure described in the item **Electron backscatter diffraction** of Section 3.2. The as-polished state of the surface has been used for the inspection. The micrographs have been acquired with the equipment described in the item **Quantitative light optical micrography** of Section 3.2. Stitching images composed by individual micrographs taken at the high magnification of 200 have been obtained in order to have a global image of the distribution of α' -martensite. Each stitching covers a region of about 7 mm along the fracture surface and 4 mm below it, see Fig. 19. The stitching's length includes the fracture surface corresponding to the final part of the fatigue pre-cracking and the total length of the crack developed in the fracture toughness tests.

6 Fracture behaviour: results

Fig. 16 shows the load versus load-line displacement record of one of the performed fracture toughness tests for AISI 304L at 4 K. The graphic also includes the intermediate points selected for the ASTM Normalization Method (see Section 5.1) –blue filled circles– as well as the points identified as the onset and end of each unloading –red star symbols–, values which are needed for the construction of the $J - R$ curve using the compliance methods (ASTM, W-N and Modified W-N Compliance Methods). Notice that, since the test is performed at 4 K, the loading curve shows serrated yielding (see Section 4) which gets more pronounced as the displacement increases. Special attention is paid during the identification of the aforementioned points so that none of the serrations is recognized as an unloading sequence, otherwise false points in the construction of the $J - R$ curve might influence the determination of the fracture toughness parameters. On the other hand, it might happen that an unloading sequence is carried out just after a serration takes place, in that case an underestimated load value for the corresponding unloading onset point is obtained (see for example the second unloading which is shown on the magnified area in Fig. 16). Nevertheless, as it is discussed in a following paragraph (see Table 4), the similarity between the results obtained with the Modified W-N Compliance Method and the ASTM Normalization Method suggests that, for the tests analysed in this work, the underestimated load of these individual points has a negligible influence on the determination of the fracture toughness parameters.

The fracture toughness experimental results for AISI 304L and AISI 316LN are shown in Fig. 17. The crack-growth resistance $J - R$ curves are computed with the ASTM Normalization Method. Filled and empty symbols represent data points that fall inside and outside the “Region of Qualified Data”, which is determined according to Fig. A9.2 of the standard ASTM-E1820-20a (2020). Large darker symbols indicate the fracture toughness J_Q , which is obtained from the intersection of the power law regression line with the “0.2 – mm offset line” (Section A9.6.6 of the standard ASTM-E1820-20a (2020)). The power law regression line is defined by fitting the expression $J = C_1 \Delta a^{C_2}$, where Δa is the crack extension and C_1 and C_2 are constants, to the data that fall into the “Region of Qualified Data”, and it is pictured with solid lines in the figure. Dashed lines correspond to the “construction line” (blunting line) and the “0.2 – mm offset line”, as indicated in the graph, with the slope being $2\sigma_Y = \sigma_{YS} + \sigma_{TS}$ (using the engineering definition of stress) according to the standard ASTM-E1820-20a (2020). Results corresponding to three different experiments (i.e., three different realizations for the same material and testing

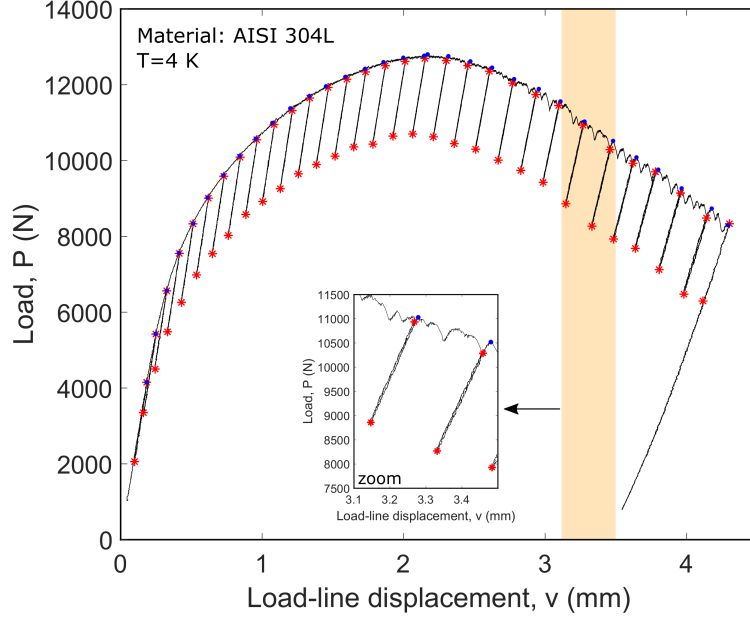


Figure 16: Load versus load-line displacement record corresponding to a fracture toughness test for AISI 304L performed at 4 K. Blue filled circles represent the individual points used for the ASTM Normalization Method. Red star symbols represent the onset and end of each unloading.

conditions) indicated with circular, square and triangular symbols are included in each graph. Specifically, Figs. 17(a) and 17(b) picture the results for AISI 304L tested at 77 K and 4 K, respectively, and Fig. 17(c) shows the results for AISI 316LN tested at 4 K. Notice that, in the three graphs, the results obtained with the three realizations are similar, which shows the robustness and repeatability of the measurements. The comparison of results for both materials and testing temperatures shows that AISI 316LN displays higher fracture toughness than AISI 304L (check Figs. 17(b) and 17(c)), and for AISI 304L the fracture toughness decreases with decreasing testing temperature (check Figs. 17(a) and 17(b)). No results for AISI 316LN at 77 K are provided as during the three performed fracture toughness tests the crack did not propagate for displacements within the clip gage measuring range, so that no data points within the “Region of Qualified Data” were obtained. The comparison of the crack-growth resistance $J - R$ curves obtained in this work with data available in the literature is performed in B.

Optical micrographs of the fracture surface for experiments performed with AISI 304L samples tested at 77 K and 4 K and AISI 316LN samples at 4 K are presented in Fig. 18. The sharp notch, the surface corresponding to the pre-cracking, and the surface corresponding to the fracture toughness test are indicated in the pictures. A close view of the blunting zone is enlarged on the left bottom part of Fig. 18(a). In the case of AISI 304L, Figs. 18(a)-(b), the crack front presents the so-called tunnelling effect (Weiss and Nyilas, 2006), with much shorter crack extension near the sample’s edges. In contrast, notice that for the AISI 316LN sample, the tunnelling effect is less pronounced. Irrespective of the different crack front shapes, the crack front of all tested samples fulfils the requirements of the standard ASTM-E1820-20a (2020) (see Sections 9.1.4.1, 9.1.4.2 and 9.1.5.1 therein). Moreover, the optical micrographs make evident the formation of a neck in the samples –pointed out in the pictures– that leads to a reduction of thickness of $\approx 5.5\%$, $\approx 3\%$ and 5% for AISI 304L tested at 77 K, AISI 304L at 4 K and AISI 316LN at 4 K, respectively. Even though the reduction of thickness is significant, the plane-strain conditions stated in the standard ASTM-E1820-20a (2020) ($B > 10J_Q/\sigma_Y$ and $b_0 > 10J_Q/\sigma_Y$ according to Sections A9.10.1 and A9.10.2) are fulfilled for all the samples tested in this work, as it is also discussed in a following paragraph. Additionally, the fracture surfaces of all tested samples show symmetry with respect to the middle-thickness plane (see Fig. 18) which together with the fact that the crack developed along the sample’s middle-height plane (see Fig. 15) indicate

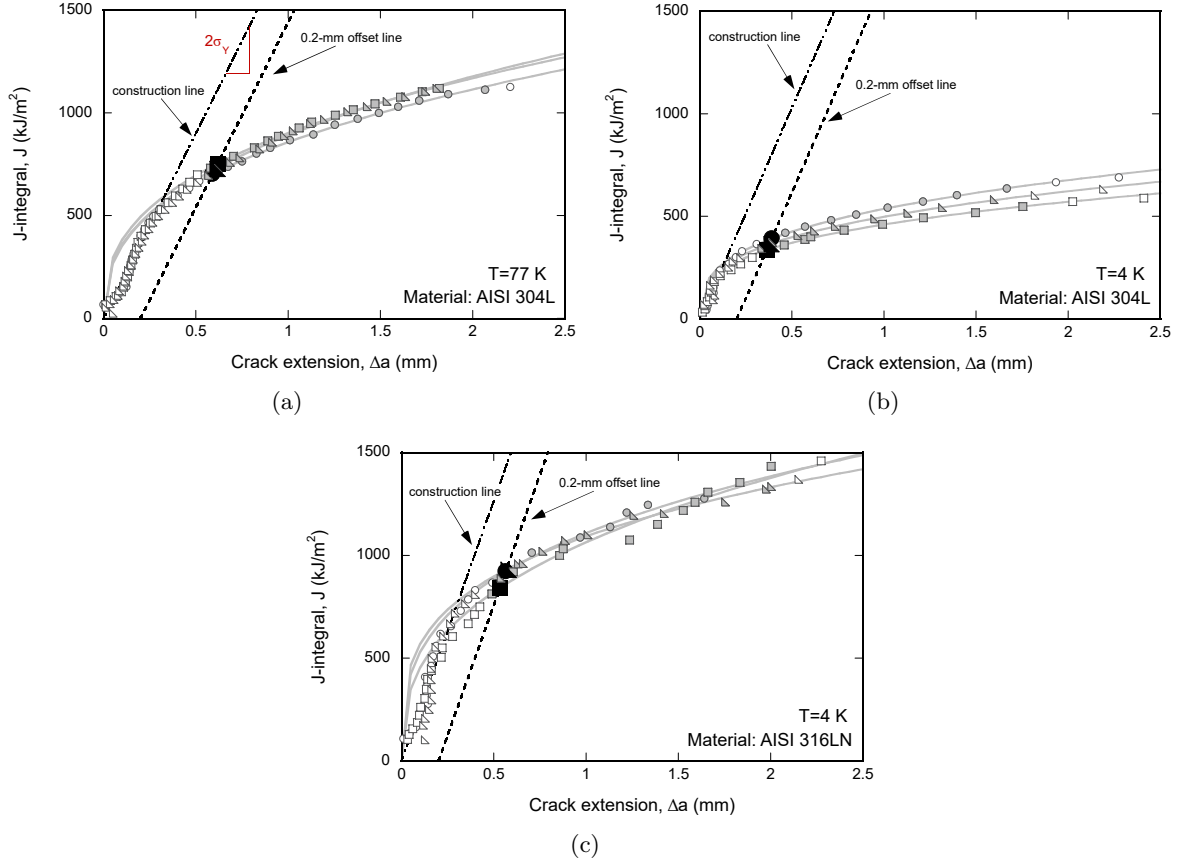


Figure 17: Fracture toughness experimental results for AISI 304L and AISI 316LN. Crack-growth resistance $J - R$ curves computed with the ASTM Normalization Method. Filled and empty symbols represent data points that fall inside and outside the “Region of Qualified Data”, respectively. Large darker symbols indicate the fracture toughness J_Q . Solid lines represent the power law regression fitting. Dashed lines correspond to the “construction line” (blunting line) and the “0.2-mm offset line”, as indicated in the graphs. Results corresponding to three different experiments indicated with circular, square and triangular symbols are included in each graph: (a) AISI 304L tested at $T = 77$ K, (b) AISI 304L tested at $T = 4$ K and (c) AISI 316LN tested at $T = 4$ K.

a good alignment and Mode I loading dominance (negligible contribution of Mode II and Mode III) during fatigue pre-cracking and fracture toughness tests.

Fig. 19 shows stitched micrographs obtained by light optical microscopy of the fractured samples near the fracture surface for the same samples investigated in Fig. 18. The surfaces corresponding to the pre-cracking and the fracture toughness test are indicated in the pictures. A close view of the fracture toughness area is enlarged on the bottom part of the images in order to illustrate the lath-type α' -martensite morphology. Note that the martensite appears in black, and the austenite in grey, as indicated in the legend located in the bottom left part of the micrographs. The highest concentration of martensite is located next to the fracture surface, decreasing towards the bulk, most likely due to decreasing plastic strain. The content of martensite seems to be greater in AISI 304L specimens, which is consistent with the results reported in Figs. 13 and 14 of Section 4, which showed that the martensitic transformation is faster and more important in AISI 304L than in AISI 316LN. Note that for AISI 304L, more martensite is observed in the sample tested at 77 K, most likely because the plastic deformation near the crack is greater than at 4 K. Furthermore, the AISI 304L specimens show more regular and plane crack path in comparison with the results obtained with AISI 316LN, as can be anticipated from Fig. 18(c) where the fracture surface of the AISI 316LN sample shows a rough and granular appearance. This can be attributed to the higher amount of the martensitic phase shown by the

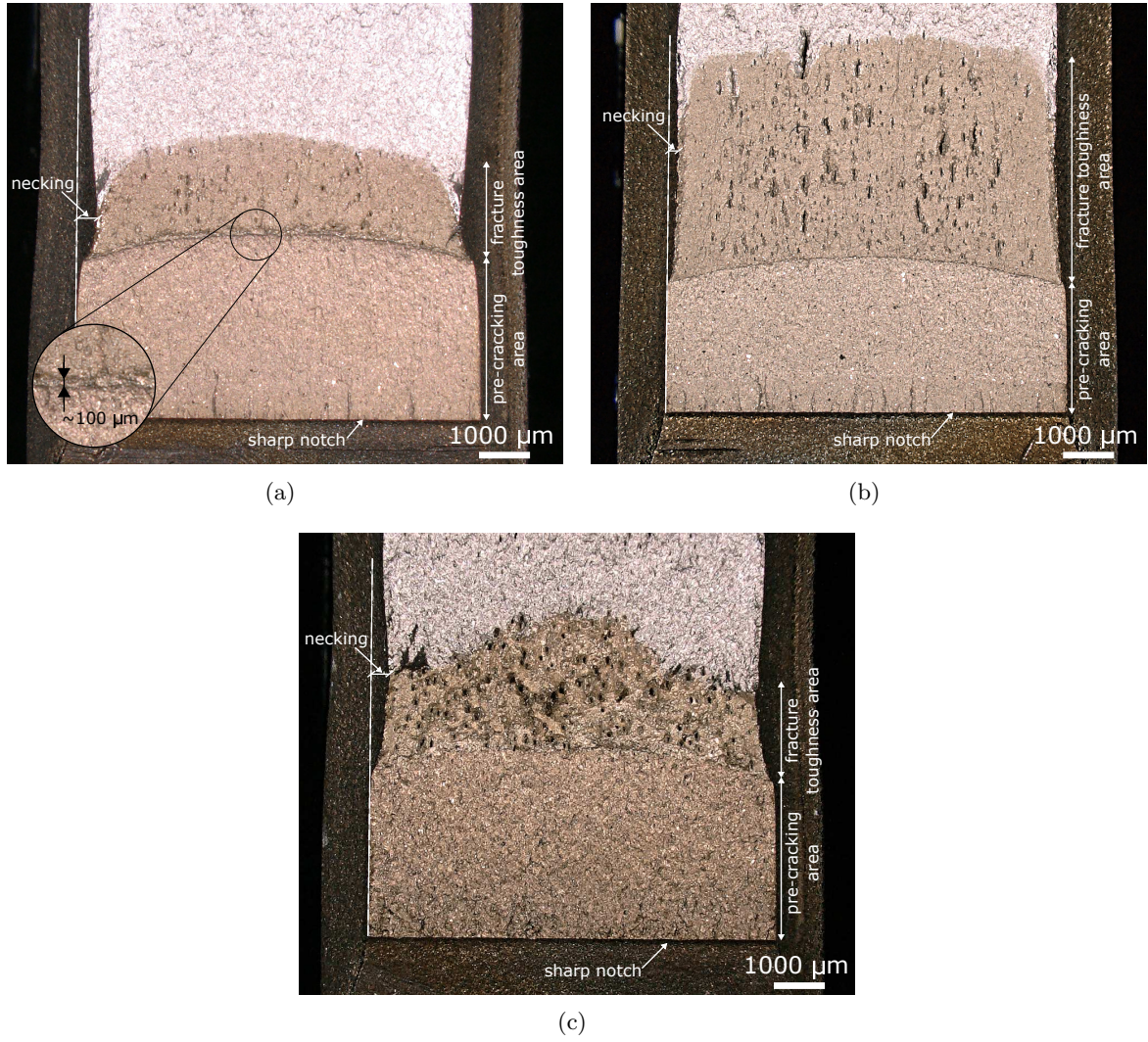


Figure 18: Optical micrographs of the fracture surface for: (a) AISI 304L tested at $T = 77$ K, (b) AISI 304L tested at $T = 4$ K and (c) AISI 316LN tested at $T = 4$ K. The neck, the sharp notch, the surface corresponding to the pre-cracking, and the surface corresponding to the fracture toughness tests are indicated in the pictures. Moreover, a close view of the blunting zone is also enlarged on the left bottom part in the picture corresponding to AISI 304L tested at $T = 77$ K.

AISI 304L specimens (the martensite being less ductile than the austenite). Moreover, Fig. 19(a) also makes evident the blunting profile in the transition between the pre-cracking area and the fracture toughness area of AISI 304L at 77 K (see Fig. 3.25(b) from Anderson (2017)), recall that the blunting is also noticeable from a top view of the fracture surface as it is shown in Fig. 18(a). Based on the experimental tests it is not possible to provide further conclusions regarding the contribution of the martensitic transformation and of the blunting to the fracture toughness results. For this purpose, the reader is referred to the finite element calculations performed by Iwamoto and Tsuta (2002) for Compact Samples subjected to Mode I loading, which evaluate the fracture toughness for an austenitic material (AISI 304) with and without martensitic transformation.

The comparison between the results obtained with the four approaches described in Section 5.1 to compute the crack-growth resistance $J - R$ curves (ASTM Compliance Method, W-N Compliance Method, Modified W-N Compliance Method and ASTM Normalization Method) is performed in Fig. 20. As in Fig. 17, filled and empty symbols represent data points that fall inside and outside the “Region of Qualified Data”, respectively. Large darker symbols indicate

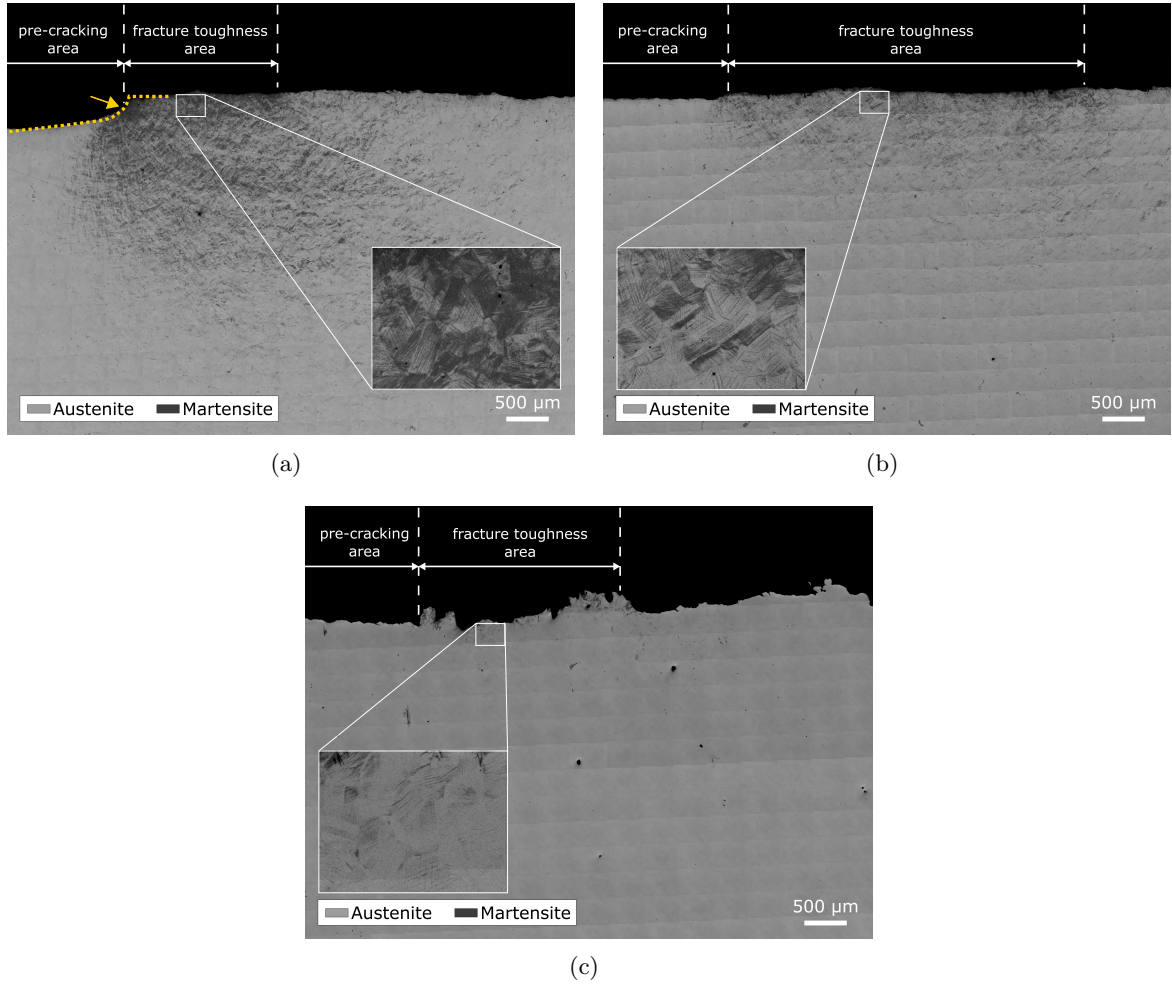


Figure 19: Stitched micrographs obtained by light optical microscopy of the fractured specimens near the fracture surface. (a) AISI 304L tested at $T = 77$ K, (b) AISI 304L tested at $T = 4$ K and (c) AISI 316LN tested at $T = 4$ K. For the image corresponding to AISI 304L tested at 77K, the crack profile in the transition between the pre-cracking area and the fracture toughness area is indicated with a yellow dashed line and the blunting is pointed out with a yellow arrow.

the fracture toughness J_Q , the power law regression fitting being represented by solid lines. Each colour corresponds to the results obtained with a given method, see the legend in the bottom right part of the graph. There are noticeable differences between the crack-growth resistance $J - R$ curves obtained with the four different methods. For instance, while the values of J computed with the Modified W-N Compliance Method and the ASTM Normalization Method are similar, they lie above the data obtained with the W-N Compliance Method. Moreover, note that, in comparison with the other three approaches, the ASTM Compliance Method greatly underestimates the value of J_Q : the negative crack extension causes two points with $J \approx 0$ to fall into the “Region of Qualified Data” so that the power law regression line does not properly represent the $J - R$ evolution obtained for most of the data points.

Additional quantitative comparison between the results obtained with the four approaches used to compute the $J - R$ curves is provided in Table. 4 which displays the stress intensity factor K_{JQ} calculated for AISI 304L at 77 K and 4 K and for 316LN at 4 K for the three experiments performed per material-temperature combination. The results are expressed in terms of the fracture toughness parameter K_{JQ} instead of J_Q as the former is widely used in the literature. Nevertheless, the same discussion for K_{JQ} (and K_{JIC}) is valid for J_Q (and J_{IC}) since, as discussed in Section 5.1, the two parameters are directly related by the formula $K_{JQ} = \sqrt{(EJ_Q)/(1 - \nu^2)}$ (and $K_{JIC} = \sqrt{(EJ_{IC})/(1 - \nu^2)}$, see Sections A9.11 of the standard ASTM-E1820-20a (2020)).

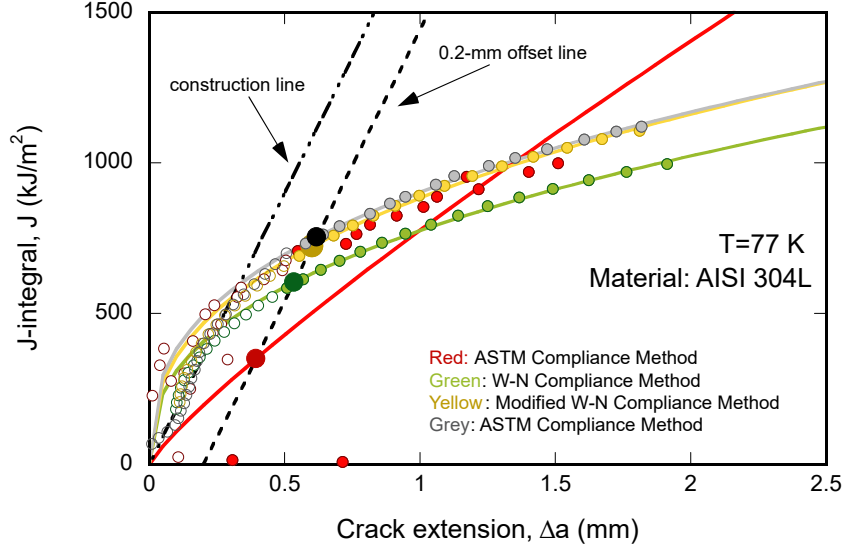


Figure 20: Fracture toughness experimental results for AISI 304L tested at $T = 77$ K. Crack-growth resistance $J - R$ curves computed with the four approaches described in Section 5.1: ASTM Compliance Method, W-N Compliance Method, Modified W-N Compliance Method and ASTM Normalization Method. Filled and empty symbols represent data points that fall inside and outside the ‘Region of Qualified Data’, respectively. Large darker symbols indicate the fracture toughness J_Q . Solid lines represent the power law regression fitting. Each colour corresponds to the results obtained with a given method. Dashed lines correspond to the “construction line” and the “0.2 – mm offset line”.

Notice that for some experiments the tests data do not fulfil the requirements established by the standard for K_{JQ} to be considered the actual stress intensity factor. To be highlighted that if K_{JQ} is the actual stress intensity factor (i.e. it fulfils all the requirements of the standard), we have that $K_{JQ} = K_{JIc}$ (size-independent plane-strain stress intensity factor), since the size criteria, $B > 10J_Q/\sigma_Y$ and $b_0 > 10J_Q/\sigma_Y$ according to Sections A9.10.1 and A9.10.2 of the standard ASTM-E1820-20a (2020), are fulfilled for all the tests performed in this work.

For instance, none of the K_{JQ} values which have been obtained using the ASTM Compliance Method fulfil all the requirements listed in ASTM-E1820-20a (2020) because the qualification requirements included in Sections A9.3.3.2, A9.4 and 9.1.5.2 and Note A9.1 are repeatedly breached. Note A9.1 indicates that *anomalies* at the beginning of the test, due to the apparent negative crack extension in the experiments performed in this work, cause points with low J to fall within the “Region of Qualified Data” providing an unreasonable low value of K_{JQ} (where Note A9.1 is mentioned in Table 4, the resulting K_{JQ} value is not included as it is not consistent with the corresponding $J - R$ curve, see Fig. 20). Furthermore, since the ASTM Compliance Method does not address the negative crack extension phenomenon, see the item **ASTM Compliance Method** in Section 5.1, the adjustment procedure to obtain an improved estimation of the initial crack size is not effective to evaluate the data obtained from most tests. Specifically, the correlation coefficient of the data which resulted from the adjustment procedure is lower than 0.96, so that the test data are invalid according to Section A9.3.3.2 of ASTM-E1820-20a (2020). In addition, for some tests the adjustment procedure underestimates the initial crack size by more than 0.5 mm with respect to the measurements obtained with optical microscopy, making the result invalid according to Section A9.4 of ASTM-E1820-20a (2020). The large difference between estimated and measured initial crack size is most likely due to the defective estimation of the crack size using the compliance equations rather than a consequence of the adjustment procedure, so that for some of the experiments the difference between predicted and measured crack extension is larger than the maximum value accepted in Section 9.1.5.2 of ASTM-E1820-20a (2020).

In contrast, the W-N Compliance Method addresses the problem related to the negative crack

extension and corrects the estimation of the crack lengths, see the item **W-N Compliance Method** in Section 5.1. Furthermore, despite the estimation of the initial crack length does not match with the measurement obtained with optical microscopy, the difference is lower than 200 μm , it is acceptable according to Section A9.4 of the standard ASTM-E1820-20a (2020) so that the values of K_{JQ} qualify as K_{JIC} .

For the Modified W-N Compliance Method all the resulting K_{JQ} values also qualify as K_{JIC} . The quantitative differences with the results obtained with the W-N Compliance Method –which yields lower values for K_{JQ} – are most likely due to the re-calculation of the values of J using the improved values of the crack sizes, see the item **Modified W-N Compliance Method** in Section 5.1. Moreover, the iterative process implemented for the estimation of the crack sizes predicts that the crack extension caused by blunting is approximately 100 μm , 30 μm and 80 μm for AISI 304L at 77 K, AISI 304L at 4 K and AISI 316LN at 4 K, respectively. Note that the predictions for AISI 304L find reasonable agreement with the experimental measurements presented in Fig. 18.

Regarding the ASTM Normalization Method, some of the tests do not comply with the additional restriction included in Section A15.2.4 of the standard ASTM-E1820-20a (2020) which states that the measured crack extension should not exceed the lesser of 4 mm or 15% of the initial uncracked ligament. Nonetheless, the other additional requirements included in the standard are met, so that the values of the stress intensity factor obtained from the tests are assumed to qualify as K_{JIC} . Furthermore, while this approach does not require to carry out a series of unloading-reloading sequences during the fracture toughness tests, which makes the ASTM Normalization Method more sound and practical to analyse as compared to the other three compliance methods (see the item **ASTM Normalization Method** in Section 5.1), the results obtained for K_{JQ} are very similar, qualitatively and quantitatively, to the ones computed with the Modified W-N Compliance Method. This also suggests that breaching the additional requirement included in Section A15.2.4 of the standard ASTM-E1820-20a (2020) does not have an important influence for the determination of K_{JQ} .

	ASTM Compliance Method		W-N Compliance Method		Modified W-N Compliance Method		ASTM Normalization Method	
	K_{JQ} (MPa√m)	Invalid requirements	K_{JQ} (MPa√m)	Invalid requirements	K_{JQ} (MPa√m)	Invalid requirements	K_{JQ} (MPa√m)	Invalid requirements
AISI 304L T = 77 K	385.56	Section A9.3.3.2 Section A9.4 Section 9.1.5.2	348.62	-	389.60	-	399.04	-
	-	Section A9.3.3.2 Section A9.4 Section 9.1.5.2	369.87	-	404.71	-	413.11	-
	367.57	Section A9.4 Section 9.1.5.2 Note A9.1	353.43	-	393.06	-	405.93	-
	-	Section A9.3.3.2 Section A9.4 Section 9.1.5.2 Note A9.1	273.31	-	286.37	-	298.03	Section A15.2.4
AISI 304L T = 4 K	264.46	Section A9.3.3.2	247.41	-	251.11	-	276.21	Section A15.2.4
	-	Section A9.3.3.2 Note A9.1	270.34	-	272.82	-	287.19	Section A15.2.4
	-	Section A9.3.3.2 Section A9.4 Section 9.1.5.2 Note A9.1	414.35	-	434.23	-	456.89	-
AISI 316LN T = 4 K	-	Section A9.3.3.2 Section A9.4 Section 9.1.5.2 Note A9.1	425.93	-	444.84	-	436.66	Section A15.2.4
	-	Section A9.3.3.2 Section A9.4 Section 9.1.5.2 Note A9.1	419.20	-	448.35	-	458.97	-
	-	Section A9.3.3.2 Section A9.4 Section 9.1.5.2 Note A9.1	419.20	-	448.35	-	458.97	-

Table 4: Stress intensity factor K_{JQ} results obtained from the fracture toughness tests (three experiments per material-temperature combination) with the four approaches described in Section 5.1 to compute the crack-growth resistance $J - R$ curves: ASTM Compliance Method, W-N Compliance Method, Modified W-N Compliance Method and ASTM Normalization Method. The requirements of the standard that are not met are indicated for each test.

7 Concluding remarks

In this paper, we have performed a comprehensive experimental characterization of the microstructural evolution and the mechanical behaviour and fracture of AISI 304L and AISI 316LN austenitic stainless steels at cryogenic temperatures. For that purpose, we have devised an experimental methodology which includes uniaxial tensile experiments and Mode I fracture tests in liquid nitrogen (77 K) and liquid helium (4 K).

We have shown that both AISI 304L and AISI 316LN display a significant increase of the flow stress with decreasing testing temperature and discontinuous plastic flow at 4 K. On the other hand, while AISI 316LN retains quasi-linear strain hardening and large ductility at cryogenic temperatures, the stress-strain characteristic of AISI 304L displays a sigmoidal shape and a drop in ductility of nearly 50% as compared to room temperature. Note that we have designed a novel Double-Section Sample to perform interrupted tensile tests in which the sample is unloaded before fracture to measure the martensite content at different strain levels. Post-mortem microstructural analysis of the tensile samples using magnetic induction, electron backscatter diffraction and quantitative light optical micrography allowed to determine the evolution of the martensitic transformation with the strain. The code developed for the determination of the content of martensite from light optical micrographs is included as *Supplementary Material*. The results obtained with the three methods show the same general trends and reasonable quantitative agreement, bringing out that the martensitic transformation in AISI 304L occurs faster and to a greater extent than in AISI 316LN both at 77 K and at 4 K. To the authors' knowledge, in this paper we provide the first experimental results for the evolution of the content of strain-induced martensite in AISI 304L and AISI 316LN samples tested at liquid helium temperature. Moreover, note that for AISI 304L the evolution of the martensite content at 77 K and 4 K is very similar, which seems to indicate that for this grade the transformation process is already saturated at 77 K, which is an original result of this paper. In addition, the experimental data for the strain-induced martensite fraction have been used to identify the temperature-dependent parameters of the transformation kinetic model proposed by Olson and Cohen (1975).

Moreover, the fracture tests data have been used to determine the crack-growth resistance $J - R$ curves and the fracture toughness of the investigated materials using four different methods here referred to as ASTM Compliance Method, W-N Compliance Method, Modified W-N Compliance Method (which is proposed in this work) and ASTM Normalization Method, which is an original methodological contribution of this paper. Out of these four approaches, only the $J - R$ curves computed with the W-N Compliance Method and the Modified W-N Compliance Method fulfil all the requirements of the standard ASTM-E1820-20a (2020) so that the calculated fracture toughness can be considered a material property. On the other hand, note that the results obtained with ASTM Normalization Method are very similar to the crack-growth resistance curves and the fracture toughness computed with the Modified W-N Compliance Method, the main advantage of the ASTM Normalization Method being the simpler procedures required for the test performance and the analysis of test data. The codes developed for the construction of the crack-growth resistance $J - R$ curves, and the identification of the fracture toughness are included as *Supplementary Material*. The experiments have shown that AISI 316LN has higher fracture toughness than AISI 304L, and for AISI 304L the fracture toughness decreases with decreasing testing temperature. The results are in good agreement with the experimental data available in the literature. In addition, post-mortem optical micrography examination of the fracture samples has shown that the amount of austenite transformed into martensite near the fracture surface is greater for AISI 304L than for AISI 316LN. On the other hand, for AISI 304L there is more martensite formed in the sample tested at 77 K because the plastic deformation near the crack is greater than at 4 K.

Acknowledgements

The authors would like to thank A. Gallifa-Terricabras (CERN) for performing the analysis with Digital Image Correlation and A. T. Pérez-Fontenla (CERN) for the help with the SEM imaging. Thanks are also owed to K. Weiss (KIT) for the helpful discussions about fracture toughness.

The research leading to these results has received funding from the European Union’s Horizon2020 Programme (Excellent Science, Marie Skłodowska-Curie Actions) under REA grant agreement 777896 (Project QUANTIFY).

Supplementary material

In the following links we provide access to the code developed for the determination of the content of martensite from light optical micrographs: <https://doi.org/10.21950/OX2ZFR>; and to the codes developed for the construction of the crack-growth resistance $J - R$ curves, and the identification of the fracture toughness: <https://doi.org/10.21950/XTQRTZ>.

A Flow behaviour: comparison with experimental data available in the literature

Figs. 21 and 22 show a comparison between the stress-strain curves obtained in this work for AISI 304L and AISI 316LN, and results for the same grades available in the literature with chemical composition given in Table 5. Note that all the experimental data for AISI 304L and AISI 316LN available in the literature showed discontinuous plastic flow at 4 K, however, we just display the upper envelop in Figs. 21(c) and 22(c) to facilitate the comparison of results. Generally, there is good qualitative and quantitative agreement between the stress-strain characteristics obtained in this work and the experimental evidence used for comparison.

Fig. 23 shows a comparison between the results for the evolution of the volume fraction of martensite $f^{\alpha'}$ with strain ε obtained in this work and experimental data available in the literature for tests performed at 300 K and 77 K for the same steel grades with chemical composition given in Table 5. Generally, there is good qualitative and quantitative agreement between the results obtained in this work and the experimental evidence used for comparison. However, no results are available in the literature for AISI 304L and AISI 316LN samples tested at 4 K so that we could compare the martensite volume fraction measurements presented in this paper. No graph is included for AISI 316LN tested at room temperature because, in agreement with other authors, see Botshekan et al. (1998) and Pattanayak (1984), the martensitic transformation is negligible.

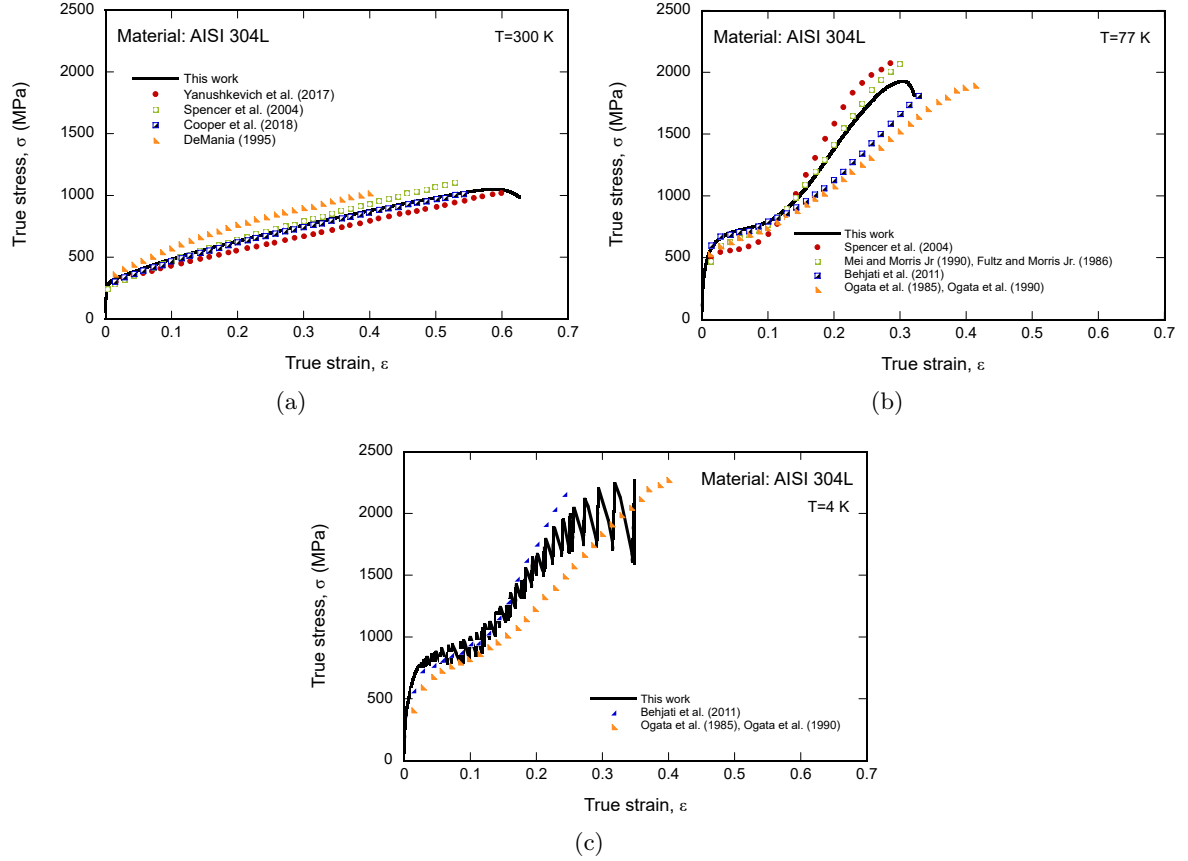


Figure 21: Comparison between the stress-strain curves obtained in this paper for AISI 304L and experimental results taken from the works of Yanushkevich et al. (2017), Spencer et al. (2004), Cooper et al. (2018), DeMania (1995), Mei and Morris Jr (1990), Fultz and Morris Jr (1986), Behjati et al. (2011), Ogata et al. (1985) and Ogata et al. (1990). Data corresponding to tensile samples tested under quasi-static loading at different temperatures: (a) $T = 300$ K, (b) $T = 77$ K and (c) $T = 4$ K.

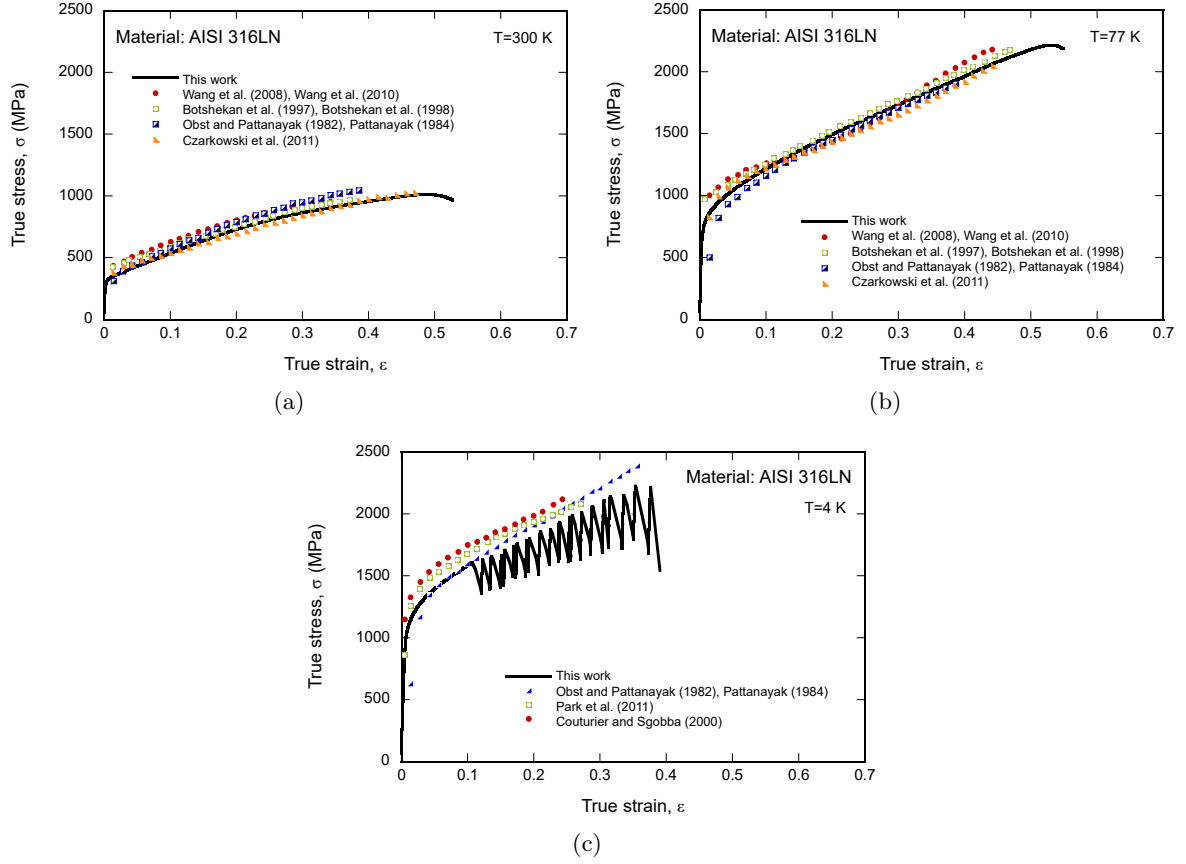


Figure 22: Comparison between the stress-strain curves obtained in this paper for AISI 316LN and experimental results taken from the works of Wang et al. (2008), Wang et al. (2010), Botshekan et al. (1997), Botshekan et al. (1998), Obst and Pattanayak (1982), Pattanayak (1984), Czarkowski et al. (2011), Park et al. (2011) and Couturier and Sgobba (2000). Data corresponding to tensile samples tested under quasi-static loading at different temperatures: (a) $T = 300$ K, (b) $T = 77$ K and (c) $T = 4$ K.

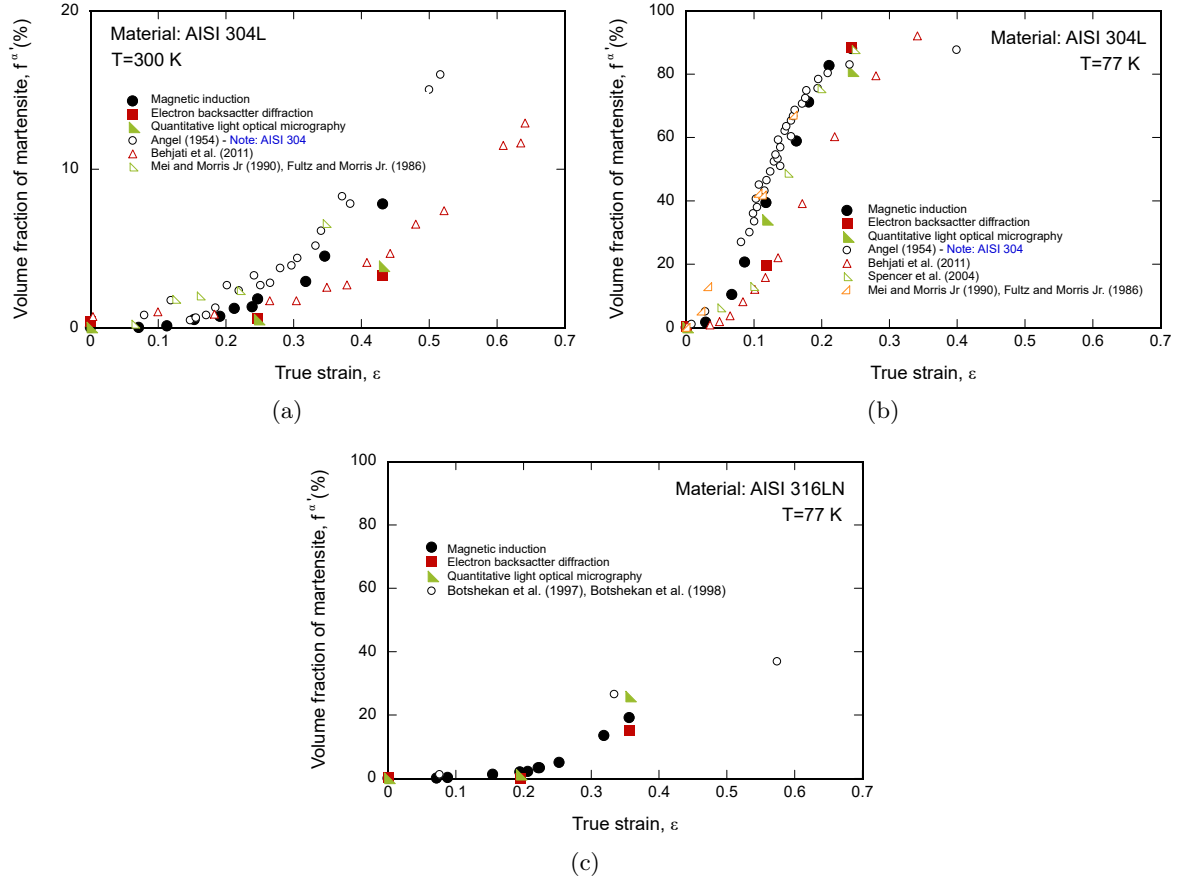


Figure 23: Comparison between the evolution of the volume fraction of martensite $f^{\alpha'}$ with strain ϵ obtained in this paper and experimental results taken from the works of Angel (1954), Behjati et al. (2011), Fultz and Morris Jr (1986), Mei and Morris Jr (1990), Spencer et al. (2004), Botshekan et al. (1997) and Botshekan et al. (1998). Data corresponding to tensile samples tested under quasi-static loading: (a) AISI 304L tested at $T = 300$ K, (b) AISI 304L tested at $T = 77$ K and (c) AISI 316LN tested at $T = 77$ K.

AISI 304L												
Source	C	S	N	Cr	Ni	Mn	Si	Mo	P	Co	Cu	Nb
Yanushkevich et al. (2017)	0.04	0.04	-	18.20	8.80	1.70	-	-	0.05	-	0.50	0.05
Spencer et al. (2004)	0.02	-	0.07	18.22	9.03	1.85	0.56	0.34	-	0.13	0.35	-
Cooper et al. (2018)	0.03	-	-	19.40	9.65	1.65	0.57	0.35	-	-	-	-
DeMania (1995)	0.02	-	0.04	18.37	9.31	1.39	0.42	0.35	0.02	-	0.39	-
Mei and Morris Jr (1990)	0.02	0.01	0.07	18.70	8.64	1.63	0.51	-	0.02	-	-	-
Fultz and Morris Jr (1986)												
Behjati et al. (2011)	0.03	0.03	-	18.00	10.00	2.00	1.00	-	0.04	-	-	-
Ogata et al. (1985)	0.02	0.01	-	18.24	10.03	1.52	0.67	-	0.03	-	-	-
Ogata et al. (1990)												
AISI 316LN												
Source	C	S	N	Cr	Ni	Mn	Si	Mo	P	Co	Cu	Nb
Wang et al. (2008)	0.01	-	0.14	15.30	12.54	1.48	0.21	2.50	0.01	-	-	-
Wang et al. (2010)												
Botshekan et al. (1997)	0.03	0.03	0.16	17.2	13.5	2.00	1.00	2.50	0.05	-	-	-
Botshekan et al. (1998)												
Obst and Pattanayak (1982)	0.03	0.02	0.16	16.70	13.70	1.26	0.41	2.68	0.02	-	-	-
Pattanayak (1984)												
Czarkowski et al. (2011)	0.02	0.01	0.15	17.10	12.60	1.15	0.53	2.66	0.07	-	0.16	-
Park et al. (2011)	0.01	-	0.16	17.27	13.55	1.70	0.15	2.56	0.02	0.02	-	-
Couturier and Sgobba (2000)	0.02	-	0.16	17.33	13.03	1.26	0.61	2.61	0.03	-	-	-

Table 5: Data source and chemical composition of the reference materials presented in Fig. 21, 22 and 23 (weight-%, Fe balance).

B Fracture behaviour: comparison with experimental data available in the literature

Figs. 24 shows a comparison between crack-growth resistance $J - R$ curves computed in this paper with the ASTM Normalization Method and experimental results taken from the works of Lee et al. (1998), Murase et al. (1993) and Nyilas and Yanagi (1989) (chemical composition given in Table 6). For the experiments performed in this work filled and empty symbols represent data points that fall inside and outside the “Region of Qualified Data”, respectively, large darker symbols indicate the fracture toughness J_Q , solid lines represent the power law regression fitting, and dashed lines correspond to the “construction line” and the “0.2 – mm offset line”. Data are shown for AISI 304L tested at 77 K and 4 K in Figs. 24(a) and 24(b), respectively, and for AISI 316LN tested at 4 K in Fig. 24(c). There is qualitative agreement between the experimental results obtained in this work and the data available in the literature –higher fracture toughness for AISI 316LN, and decreasing fracture toughness with decreasing testing temperature for AISI 304L–, and also quantitative agreement for AISI 304L tested at 4 K and for AISI 316LN tested at 4 K.

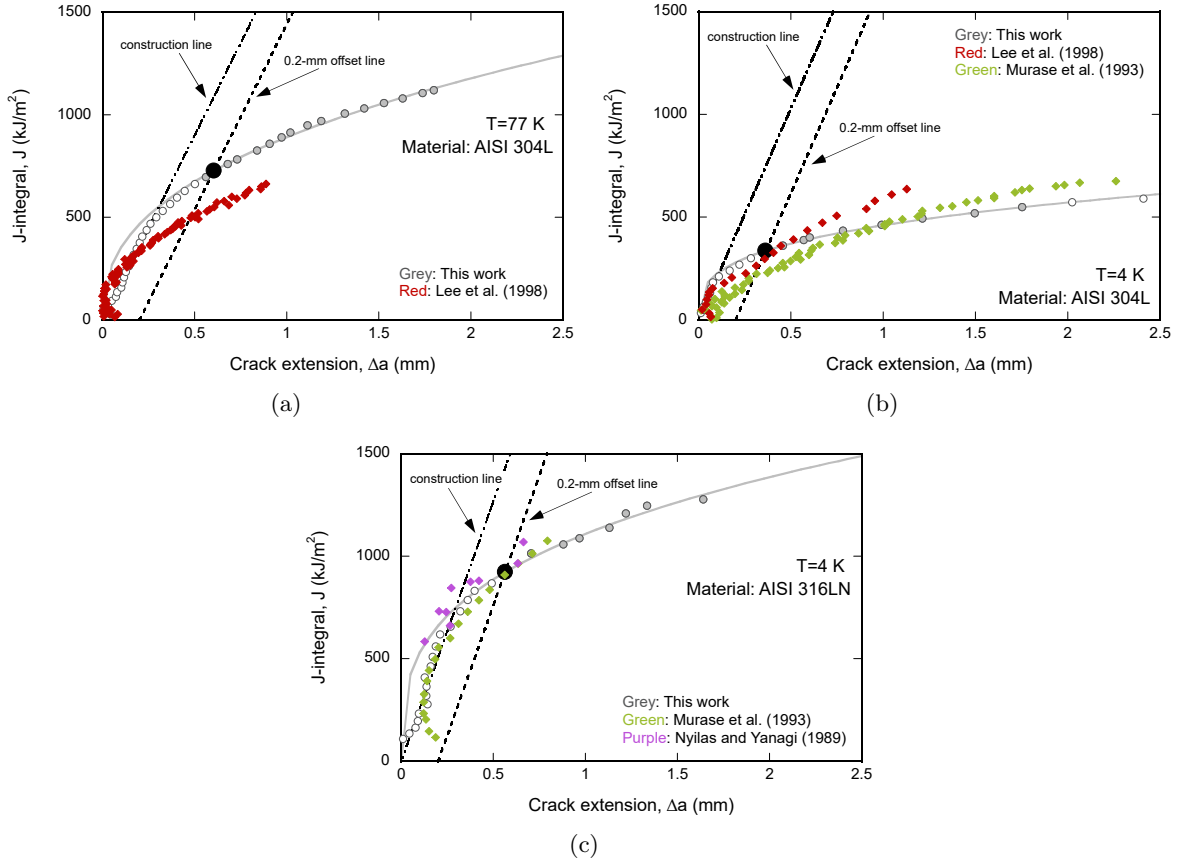


Figure 24: Comparison between crack-growth resistance $J - R$ curves computed in this paper with the ASTM Normalization Method and experimental results taken from the works of Lee et al. (1998), Murase et al. (1993) and Nyilas and Yanagi (1989). For the experiments performed in this work filled and empty symbols represent data points that fall inside and outside the “Region of Qualified Data”, respectively, large darker symbols indicate the fracture toughness J_Q , solid lines represent the power law regression fitting, and dashed lines correspond to the “construction line” and the “0.2 – mm offset line”. Data corresponding to fracture toughness tests: (a) AISI 304L tested at $T = 77$ K, (b) AISI 304L tested at $T = 4$ K and (c) AISI 316LN tested at $T = 4$ K.

Fig. 25 shows the evolution of the fracture toughness K_{JIC} with the initial 0.2% offset yield strength of the material σ_{YS} for the tests presented in this paper (solid markers). The experimental results are compared with data available in the literature for AISI 304L and AISI 316LN tested at 77 K and 4 K, and with the NIST trend line for 300 series austenitic stainless steels at 4 K taken from McRae et al. (2017). The general trend is that the fracture toughness decreases as the yield strength of the material increases, the data obtained in this paper being in reasonable qualitative agreement with the experiments taken from the literature.

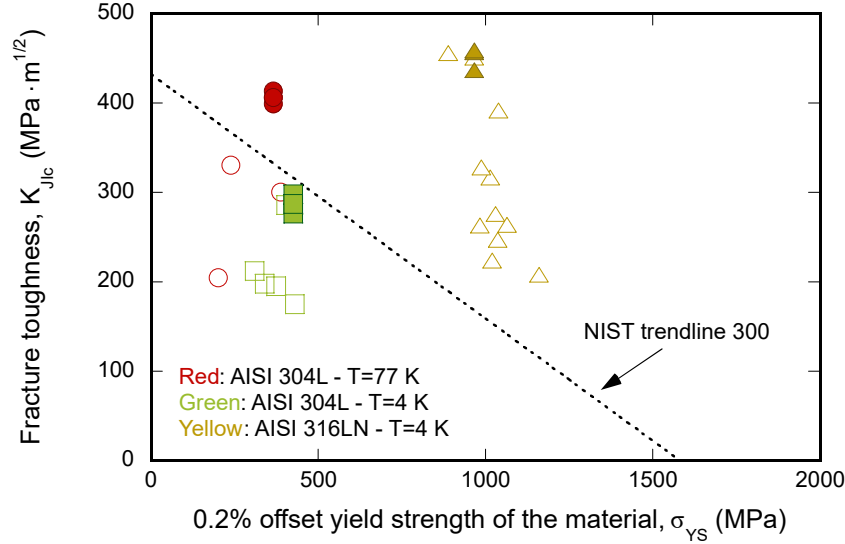


Figure 25: Fracture toughness K_{JIC} versus 0.2% offset yield strength of the material σ_{YS} . Comparison of the results obtained in this work for AISI 304L and AISI 316LN with experimental data taken from the works of Shimada et al. (1987), Lee et al. (1998), Purtscher and Read (1987), Park et al. (2019), Morris Jr et al. (1992), Murase et al. (1993), Nyilas and Mitterbacher (2010), Chan et al. (1994), Drexler et al. (1994), Krauth and Nyilas (1980), Mazandarany et al. (1980), Muster and Elster (1990), Nyilas and Yanagi (1989) and Ogata et al. (1992). The experiments taken from the literature and the results obtained in this work are represented with empty and filled symbols, respectively. The dashed line corresponds to the NIST trend line for 300 series austenitic stainless steels at 4 K, taken from McRae et al. (2017).

AISI 304L												
Source	C	S	N	Cr	Ni	Mn	Si	Mo	P	Co	Cu	Nb
Lee et al. (1998)	0.01	-	-	18.22	9.15	1.19	0.42	-	0.03	-	-	-
Murase et al. (1993)	0.02	-	-	18.36	9.67	0.82	-	-	-	-	-	-
AISI 316LN												
Source	C	S	N	Cr	Ni	Mn	Si	Mo	P	Co	Cu	Nb
Murase et al. (1993)	0.02	-	0.18	17.88	11.16	0.84	-	2.62	-	-	-	-
Nyilas and Yanagi (1989)	0.02	0.02	0.17	17.60	12.60	1.60	0.24	3.03	0.03	-	-	-

Table 6: Data source and chemical composition of the reference materials presented in Fig. 24 (weight-%, Fe balance).

References

- Anderson, T.L., 2017. Fracture mechanics: fundamentals and applications. CRC press.
- Angel, T., 1954. Formation of martensite in austenitic stainless steels. *Journal of the Iron Steel Institute* 177, 165–174.
- Apollinari, G., Béjar Alonso, I., Brüning, O., Lamont, M., Rossi, L., 2015. High-Luminosity Large Hadron Collider (HL-LHC): Preliminary design report. Technical Report. CERN, Switzerland.
- ASTM-A370-15, 2015. Standard Test Methods and Definitions for Mechanical Testing of Steel Products.
- ASTM-E112-13, 2013. Standard Test Methods for Determining Average Grain Size.
- ASTM-E1820-20a, 2020. Standard Test Method for Measurement of Fracture Toughness.
- ASTM-E1823, 2020. Standard Terminology Relating to Fatigue and Fracture Testing.
- ASTM-E399, 2020. Standard Test Method for Linear-Elastic Plane-Strain Fracture Toughness of Metallic Materials.
- ASTM-E407-07, 2015. Standard practice for microetching metals and alloys.
- ASTM-E8/E8M-15a, 2015. Standard Test Methods for Tension Testing of Metallic Materials.
- Avilés-Santillana, I., Betemps, R., Gerardin, A., Guinchard, M., Langeslag, S.A.E., Sgobba, S., 2015. Design and fabrication of a cryostat for low temperature mechanical testing for the Mechanical and Materials Engineering group at CERN. *IOP Conference Series: Materials Science and Engineering* 102.
- Barabash, V., The ITER International Team, Peacock, A., Fabritsiev, S., Kalinin, G., Zinkle, S., Rowcliffe, A., Rensman, J.W., Tavassoli, A., Marmy, P., et al., 2007. Materials challenges for ITER – Current status and future activities. *Journal of Nuclear Materials* 367, 21–32.
- Behjati, P., Najafzadeh, A., Kermanpur, A., 2011. Microstructural investigation on strengthening mechanisms of AISI 304L austenitic stainless steel during cryogenic deformation. *Materials Science and Technology* 27, 1828–1832.
- Bertinelli, F., Fudanoki, F., Komori, T., Peiro, G., Rossi, L., 2006. Production of austenitic steel for the LHC superconducting dipole magnets. *IEEE Transactions on Applied Superconductivity* 16, 1773–1776.
- Botshekan, M., Degallaix, S., Desplanques, Y., 1997. Influence of martensitic transformation on the low-cycle fatigue behaviour of 316LN stainless steel at 77 K. *Materials Science and Engineering: A* 234, 463–466.
- Botshekan, M., Degallaix, S., Desplanques, Y., Polak, J., 1998. Tensile and LCF properties of AISI 316LN SS at 300 and 77 K. *Fatigue & Fracture of Engineering Materials & Structures* 21, 651–660.
- Brüning, O., Burkhardt, H., Myers, S., 2012. The large hadron collider. *Progress in Particle and Nuclear Physics* 67, 705–734.
- Cao, B., Iwamoto, T., 2017. An experimental study on strain rate sensitivity of strain-induced martensitic transformation in SUS304 by Real-time measurement of relative magnetic permeability. *Steel Research International* 88.

- Cao, B., Iwamoto, T., Bhattacharjee, P.P., 2020. An experimental study on strain-induced martensitic transformation behavior in SUS304 austenitic stainless steel during higher strain rate deformation by continuous evaluation of relative magnetic permeability. *Materials Science and Engineering: A* 774.
- Chakrabarti, A., 2006. *Steel making*. PHI Learning Pvt. Ltd.
- Chan, J., Chu, D., Tseng, C., Morris, J., 1994. Cryogenic fracture behavior of 316LN in magnetic fields up to 14.6 T, in: *Advances in Cryogenic Engineering Materials*. Springer, pp. 1215–1221.
- Cooper, A.J., Brayshaw, W.J., Sherry, A.H., 2018. Effect of temperature on the fracture toughness of hot isostatically pressed 304L stainless steel. *Metallurgical and Materials Transactions A* 49, 811–816.
- Couturier, K., Sgobba, S., 2000. Phase stability of high manganese austenitic steels for cryogenic applications. Technical Report. CERN, Switzerland.
- Czarkowski, P., Krawczynska, A., Slesinski, R., Brynk, T., Budniak, J., Lewandowska, M., Kurzydowski, K., 2011. Low temperature mechanical properties of 316L type stainless steel after hydrostatic extrusion. *Fusion Engineering and Design* 86, 2517–2521.
- De, A.K., Murdock, D.C., Mataya, M.C., Speer, J.G., Matlock, D.K., 2004. Quantitative measurement of deformation-induced martensite in 304 stainless steel by X-ray diffraction. *Scripta Materialia* 50, 1445–1449.
- DeMania, D.A., 1995. The influence of martensitic transformation on the formability of 304L stainless steel sheet. Ph.D. thesis. Massachusetts Institute of Technology, MA, United States.
- Devred, A., Backbier, I., Besette, D., Bevilard, G., Gardner, M., Jong, C., Lillaz, F., Mitchell, N., Romano, G., Vostner, A., 2014. Challenges and status of ITER conductor production. *Superconductor Science and Technology* 27.
- Drexler, E., Simon, N., Reed, R., 1994. Strength and toughness at 4 K of forged, heavy-section 316LN, in: *Advances in Cryogenic Engineering Materials*. Springer, pp. 1199–1206.
- EN-10088-2, 2008. Stainless steels - Part 2: Technical delivery conditions for sheet/plate and strip of corrosion resisting steels for general purposes.
- FERITSCOPE-FMP30, 2008. Operator Manual. Helmut Fischer GmbH Institut für Elektronik und Messtechnik, Sindelfingen Germany.
- Fultz, B., Morris Jr, J., 1986. Effects of high magnetic fields on the flow stress of 18-8 stainless steels. *Acta Metallurgica* 34, 379–384.
- Herrera, R., Landes, J., 1990. Direct JR curve analysis: a guide to the methodology, in: *Fracture mechanics: twenty-first symposium*, ASTM International.
- ISO-6892-1, 2009. Metallic materials-Tensile testing-Part 1: Method of test at room temperature.
- ISO-6892-3, 2015. Metallic materials-Tensile testing-Part 3: Method of test at low temperature.
- ISO-6892-4, 2015. Metallic materials-Tensile testing-Part 4: Method of test in liquid helium.
- Iwamoto, T., Tsuta, T., 2002. Computational simulation on deformation behavior of ct specimens of trip steel under mode I loading for evaluation of fracture toughness. *International Journal of Plasticity* 18, 1583–1606.
- Jin, Y., Bernacki, M., Rohrer, G.S., Rollett, A.D., Lin, B., Bozzolo, N., 2013. Formation of annealing twins during recrystallization and grain growth in 304L austenitic stainless steel, in: *Materials Science Forum*, Trans Tech Publications Ltd. pp. 113–116.

- Joyce, J.A., 2001. Analysis of a high rate round robin based on proposed annexes to ASTM E 1820. *Journal of Testing and Evaluation* 29, 329–351.
- Joyce, J.A., Tregoning, R.L., 2000. Development of consistent size criteria for ASTM combined fracture mechanics standards, in: *Fatigue and Fracture Mechanics: 30th Volume*. ASTM International.
- Krauth, H., Nyilas, A., 1980. Fracture toughness of nitrogen strengthened austenitic steels at 4 K, in: *Fracture and Fatigue*. Elsevier, pp. 119–128.
- Kujanpaa, V., Suutala, N., Takalo, T., Moisio, T., 1979. Correlation between solidification cracking and microstructure in austenitic and austenitic-ferritic stainless steel welds. *Welding Research International* 9, 55–75.
- Kvackaj, T., Rozsypalova, A., Kocisko, R., Bidulska, J., Petrousek, P., Vlado, M., Pokorný, I., Sas, J., Weiss, K.P., Duchek, M., et al., 2020. Influence of processing conditions on properties of AISI 316LN steel grade. *Journal of Materials Engineering and Performance* 29, 1509–1514.
- Landes, J., Zhou, Z., Lee, K., Herrera, R., 1991. Normalization method for developing J-R curves with the LMN function. *Journal of testing and evaluation* 19, 305–311.
- Lee, H., Shin, J., Shin, H., 1998. Fracture toughness of structural materials for LNG storage tank at cryogenic temperatures. *한국가스학회: 학술대회논문집*, 340–349.
- Lee, K., 1995. Elastic-plastic fracture toughness determination under some difficult conditions. Ph.D. thesis. University of Tennessee-Knoxville, TN, United States.
- Malik, N., 2021. Development of a universal cryogenic test facility. Master's thesis. Delft University of Technology, The Netherlands.
- Margonon, P., Thomas, G., 1970. The martensite phases in 304 stainless steel. *Metallurgical Transactions* 1.
- Mazandarany, F., Parker, D., Koenig, R., Read, D., 1980. A nitrogen-strengthened austenitic stainless steel for cryogenic magnet structures, in: *Advances in Cryogenic Engineering Materials*. Springer, pp. 158–170.
- McRae, D., Balachandran, S., Walsh, R., 2017. Fatigue and fracture of three austenitic stainless steels at cryogenic temperatures, in: *IOP Conference Series: Materials Science and Engineering*.
- Mei, Z., Morris Jr, J.W., 1990. Influence of deformation-induced martensite on fatigue crack propagation in 304-type steels. *Metallurgical Transactions A* 21, 3137–3152.
- Mitchell, N., Bessette, D., Gallix, R., Jong, C., Knaster, J., Libeyre, P., Sborchia, C., Simon, F., 2008. The ITER magnet system. *IEEE Transactions on Applied Superconductivity* 18, 435–440.
- Morris Jr, J.W., Chan, J.W., Mei, Z., 1992. The influence of deformation-induced martensite on the cryogenic behavior of 300-series stainless steels. Technical Report. Lawrence Berkeley Lab., CA, United States.
- Murase, S., Kobatake, S., Tanaka, M., Tashiro, I., Horigami, O., Ogiwara, H., Shibata, K., Nagai, K., Ishikawa, K., 1993. Effects of a high magnetic field on fracture toughness at 4.2 K for austenitic stainless steels. *Fusion Engineering and Design* 20, 451–454.
- Muster, W., Elster, J., 1990. Low temperature embrittlement after ageing stainless steels. *Cryogenics* 30, 799–802.

- Naraghi, R., 2009. Martensitic transformation in austenitic stainless steels. Master's thesis. Royal Institute of Technology, Stockholm, Sweden.
- Nyilas, A., Mitterbacher, H., 2010. Fracture mechanical measurements with commercial stainless steels at 4 K and with CP-titanium at 173 K, in: AIP Conference Proceedings, American Institute of Physics. pp. 33–40.
- Nyilas, A., Obst, B., Harries, D., 1998. Mechanical investigations on aged stainless steel and Incoloy 908 materials at 4.2 K and 7 K, in: Advances in Cryogenic Engineering Materials. Springer, pp. 17–24.
- Nyilas, A., Yanagi, H., 1989. 4 K fracture toughness investigations of 316LN stainless steel plate and forging materials. Cryogenics 29, 191–195.
- Obst, B., Nyilas, A., 1991. Experimental evidence on the dislocation mechanism of serrated yielding in FCC metals and alloys at low temperatures. Materials Science and Engineering: A 137, 141–150.
- Obst, B., Pattanayak, D., 1982. Discontinuous deformation modes of a nitrogen-stabilized austenitic steel, in: Advances in Cryogenic Engineering Materials. Springer, pp. 57–65.
- Ogata, T., Ishikawa, K., Nagai, K., 1985. Effects of strain rate on the tensile behavior of stainless steels, copper, and an aluminium alloy at cryogenic temperatures. Tetsu-to-Hagane 71, 1390–1397.
- Ogata, T., Nagai, K., Ishikawa, K., Shibata, K., Murase, S., 1992. VAMAS second round robin test of structural materials at liquid helium temperature, in: Materials. Springer, pp. 69–76.
- Ogata, T., Umezawa, O., Ishikawa, K., 1990. Low temperature creep behaviour of stainless steels. Advances in Cryogenic Engineering: Materials 36, 1233–1239.
- Olson, G., Cohen, M., 1975. Kinetics of strain-induced martensitic nucleation. Metallurgical Transactions A 6.
- Ortwein, R., 2015. Constitutive model of graded micro-structure obtained via strain induced phase transformation. Ph.D. thesis. Cracow University of Technology, Poland.
- Park, J., Lee, K., Sung, H., Kim, Y.J., Kim, S.K., Kim, S., 2019. J-integral fracture toughness of high-Mn steels at room and cryogenic temperatures. Metallurgical and Materials Transactions A 50, 2678–2689.
- Park, S.H., Kim, J.Y., Park, W.W., Choi, H., Ma, Y.J., Kwon, S.P., Kim, K., Kang, S.C., Lee, D.H., 2011. The effect of plastic deformation on low temperature mechanical and magnetic properties of austenite 316LN tube for ITER TF conductor. IEEE Transactions on Applied Superconductivity 22.
- Pattanayak, D., 1984. The influence of prestrain and transformation mode on the mechanical properties of nitrogen stabilized austenitic steel, in: Advances in Cryogenic Engineering Materials. Springer, pp. 227–236.
- Pham, H.T., Iwamoto, T., 2015. An experimental investigation on rate sensitivity of fracture-mechanical characteristics in 304 austenitic stainless steel under bending deformation. ISIJ International 55, 2661–2666.
- Pham, H.T., Iwamoto, T., 2018. An evaluation of fracture properties of type-304 austenitic stainless steel at high deformation rate using the small punch test. International Journal of Mechanical Sciences 144, 249–261.

- Purtscher, P., Read, D., 1987. Effect of prior deformation on the 76-K fracture toughness of AISI 304L and AWS 308L stainless steels. *ASME Journal of Engineering Materials and Technology* 109, 151–156.
- Reed, R.P., 1989. Nitrogen in austenitic stainless steels. *The Journal Of The Minerals, Metals & Materials Society* 41, 16–21.
- Romano, G., Vostner, A., Bessette, D., Pong, I., Bevilard, G., Zhou, C., Devred, A., 2016. Mechanical properties of ITER coil CICC steel jackets production. *IEEE Transactions on Applied Superconductivity* 26, 1–4.
- Rosenthal, Y.A., Tobler, R.L., Purtscher, P.T., 1990. J_{Ic} data analysis methods with a “negative crack growth” correction procedure. *Journal of Testing and Evaluation* 18, 301–304.
- Sa, J.W., Kim, H.K., Choi, C.H., Kim, H.T., Hong, K.H., Park, H.K., Bak, J.S., Lee, K.W., Ha, E.T., 2005. Mechanical characteristics of austenitic stainless steel 316LN weldments at cryogenic temperature, in: 21st IEEE/NPS Symposium on Fusion Engineering SOFE 05, IEEE. pp. 1–4.
- Seeger, A., 1957. The mechanism of glide and work hardening in face-centered cubic and hexagonal close-packaged metals, in: *Dislocations and Mechanical Properties of Crystals*, Wiley, New York. pp. 243–325.
- Seetharaman, V., 1984. Deformation and martensitic transformation. *Bulletin of Materials Science* 6, 703–716.
- Seok, C.S., 2000. Correction methods of an apparent negative crack growth phenomenon. *International Journal of Fracture* 102, 259–269.
- Sgobba, S., 2006. Materials for high vacuum technology, an overview. Technical Report. CERN, Switzerland.
- Shimada, M., Ogawa, R., Horiuchi, T., 1987. Effects of aging on cryogenic mechanical properties of low carbon austenitic stainless steels. *Journal of Cryogenics and Superconductivity Society of Japan* 22, 265–271.
- Singh, C.D., 1996. Development of hot band annealing texture in austenitic stainless steel. *Textures and Microstructures* 28, 47–69.
- Skoczeń, B., 2001. Stability, fatigue and optimization of thin-walled structures under cryogenic conditions: Application in the structural design of colliders and cryogenic transfer lines. CERN-2001-001, CERN.
- Spencer, K., Embury, J.D., Conlon, K.T., Véron, M., Bréchet, Y., 2004. Strengthening via the formation of strain-induced martensite in stainless steels. *Materials Science and Engineering: A* 387, 873–881.
- Tabin, J., Skoczeń, B., Bielski, J., 2016. Strain localization during discontinuous plastic flow at extremely low temperatures. *International Journal of Solids and Structures* 97, 593–612.
- Tabin, J., Skoczen, B., Bielski, J., 2019. Discontinuous plastic flow coupled with strain induced fcc-bcc phase transformation at extremely low temperatures. *Mechanics of Materials* 129, 23–40.
- Tarafder, M., Tarafder, S., 2006. Modelling of Crack-tip Blunting using Finite Element Method (FEM) (GAP-0088). Technical Report. National Metallurgical Laboratory, Jamshedpur, India.

- Vander Voort, G.F., Lampman, S.R., Sanders, B.R., Anton, G.J., Polakowski, C., Kinson, J., Muldoon, K., Henry, S.D., Scott Jr, W.W., 2004. ASM Handbook. Volume 9: Metallography and Microstructures. ASM International, Materials Park, OH, United States.
- Vogt, J., Foct, J., Regnard, C., Robert, G., Dhers, J., 1991. Low-temperature fatigue of 316L and 316LN austenitic stainless steels. *Metallurgical Transactions A* 22, 2385–2392.
- Voss, B., 1983. On the problem of “negative crack growth” and “load relaxation” in single specimen partial unloading compliance tests, in: *Ductile fracture test methods*.
- Wang, S., Yang, K., Shan, Y., Li, L., 2008. Plastic deformation and fracture behaviors of nitrogen-alloyed austenitic stainless steels. *Materials Science and Engineering: A* 490, 95–104.
- Wang, W., Yan, W., Yang, K., Shan, Y., Jiang, Z., 2010. Temperature dependence of tensile behaviors of nitrogen-alloyed austenitic stainless steels. *Journal of Materials Engineering and Performance* 19, 1214–1219.
- Wang, Y., Wang, W., Zhang, B., Li, C.Q., 2020. A review on mixed mode fracture of metals. *Engineering Fracture Mechanics* 235.
- Weiss, K., Nyilas, A., 2006. Specific aspects on crack advance during J-test method for structural materials at cryogenic temperatures. *Fatigue & Fracture of Engineering Materials & Structures* 29, 83–92.
- Yanushkevich, Z., Dobatkin, S., Belyakov, A., Kaibyshev, R., 2017. Hall-Petch relationship for austenitic stainless steels processed by large strain warm rolling. *Acta Materialia* 136, 39–48.
- Zhu, X.K., Joyce, J.A., 2012. Review of fracture toughness (G, K, J, CTOD, CTOA) testing and standardization. *Engineering Fracture Mechanics* 85, 1–46.