

Heat treatment effects on three-dimensional morphology of chromium carbide in AISI D2 tool steel studied by serial sectioning method

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Abstract

We developed a three-dimensional microstructure analysis system based on the manual serial sectioning method to evaluate the heat treatment effects on chromium carbide size and morphology in AISI D2 tool steel. Two heat treatment factors, austenitizing and tempering temperature were investigated. The results show that increasing in austenitizing temperature leads to further carbide precipitation after the tempering process and increasing in the tempering temperature causes precipitate smaller secondary carbides even more than the primary carbides.

Keywords: Three-dimensional serial sectioning, Heat treatment, Tool steel, Microstructure.

1. Introduction

Currently, by the increasing progress of materials science and engineering for the synthesis engineering materials with specific applications and properties, the need for using computer simulation is felt more than ever. For this purpose, different techniques have been proposed for the computer simulation. According to the material properties which are directly related to their microstructure, different methods for simulating the microstructure of engineering materials have been emerged. One of the unique and relatively new techniques in the past 20 to 25 years, due to the growth of high-speed computers, was developed and can be worn to investigate the microstructure of the materials, named the three-dimensional method. Since all the conventional microanalysis techniques such as optical microscopy

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(OM), scanning electron microscope (SEM) and transmission electron microscope (TEM) provide only two-dimensional image of the microstructure of the matter at different scales, in all the ways in which the three-dimensional microstructure of the material should be examined, such as serial sectioning method and x-ray computed tomography, the main aim is to minimize the defects and errors in measurement of these parameters: spatial distribution, morphologies, crystallographic direction distributions and continuity of the particles and phases [1]. One of the classic examples of two-dimensional microstructure analysis failure was shown in Figure 1 [2]. In Figure 1, the two-dimensional microstructure of (a) can be constructed by three-dimensional microstructures of (b) and (c). While we know that the topology of three-dimensional spatial structures (b) and (c) are very different.

According to the materials science and engineering, using the two-dimensional microstructure studies, has achieved many successes, clearly the methods of 3D microstructure analysis are not required in all cases. Quantitative metallographic analysis methods, in most cases, can interpret successfully the relation between two-dimensional and three-dimensional microstructure of the materials. This interpretation was achieved by measuring the volumetric, surface and volumetric surface density of the phases. For reviews of many metallurgical phenomena, two-dimensional microstructure imaging can meet all the quantities mentioned above. So when the third dimension is important in the analysis of the quantitative metallography? Wu et al. [3] have shown that in the case of isotropic growth in three dimensions, grains with less than 39 surfaces shrink, and more than 39 surfaces grow. Thus, the two-dimensional cross-section does not show the actual geometry of the grains and may cause errors in the evaluation of the kinetics of grain growth. This is certainly one of the cases in which the three-dimensional structure of the grain or phase is required. Another phenomenon that requires the analysis of three-dimensional microstructures is, investigating the continuity of a second phase in metallic alloys. Reviews by Kostenko et al. [4] have shown that the cementite carbide phase has to network continuity in low carbon steels and heat treatments such as spheroidization also affect the morphology and continuity of them. In recent years, many researches have been done in the field of three-dimensional metallography for studying the

microstructure of steels [4-17]. This attention is due to the steels which are known as a widely used material in various industries like automotive, aerospace, construction, etc. In the previous studies, various methods for studying the three-dimensional microstructure of the steels have been proposed, such as electron back scattered diffraction method (EBSD), focused ion beam (FIB), scanning X-ray diffraction method, and the method of automatic or manual serial sectioning and photography with an optical microscope. In all the methods mentioned, except serial sectioning, the sample preparation is very time consuming, complex and costly. Hence, in recent years, much interest has been given to the method of serial sectioning. In this method, the sample preparation is like for the conventional metallographic methods and often includes and restricted to sanding, polishing and etching the sample by a proper solution.

Clearly, the microstructure, such as volumetric density, size, continuity and morphology of carbides could affect the mechanical properties of steel. In this research, the effect of heat treatment, due to the above factors on the mechanical properties of tool steel by 3D serial sectioning method is reviewed and a model is based on 2D and 3D quantitative metallography to evaluate the metallurgical and microstructural factors presented.

2. Materials and Methods

2.1 *Material*

In this research, to study the effect of the heat treatment on tool steel by 3D metallographic method, AISI D2 tool steel was selected, and the chemical composition is given in Table 1.

Tool steel AISI D2, because of its high hardness and hardenability and good toughness after heat treatment, has a wide-range application in the manufacturing of high pressure tools such as punches, dies and cutting tools.

2.2 *Serial Sectioning Method*

Three-dimensional metallographic method that used physical exfoliation, such as mechanical, chemical and electrochemical polishing to reduce the thickness of the sample, is called the destructive method. The most important example of this approach is the serial sectioning. This method of sample preparation, due to simplicity, low cost and relatively low processing equipment has been used in recent years.

In this method, the conventional method of sample mounting is used and thereafter, sanding and mechanical polishing the surface of the sample is done to get mirror like surface. In order to recover the 3D volume of the desired structure, initial stage is to place a marker sign on the sample surface in order to find the preliminary observed area under the microscope after the serial polishing in each step. These symptoms can be a small hole about 1 mm or indenter marker of the hardness tester on the surface of the sample. In Figure 2 [18], the equipment required for the serial sectioning procedure is shown schematically.

One of the crucial factors in 3D metallography is, imaging from a fixed specific area of the sample. Therefore, many methods for fixing the sample on the microscope and marking the sample surface have been developed. One of the new methods developed and used in this study is, place the samples were mounted inside a threaded metal flange, and locating it within the sheet which is placed on the microscope in front of the lens, that has been shown in Figure 3. Using the system that has been shown in Figure 3, at each step of sectioning, displacement and rotation of the sample during placement and the removal is prevented.

It should be noted that the methods of three-dimensional metallography have different accuracy and smallest visible scale. In the present study, the rate of sectioning is about 1.5 micrometers and 13 sections are done for each sample. The solution used to etch the entire samples is 4% solution Picral. This solution etches only the carbides and retained austenite. 11 samples of steel AISI D2 with diameter 15 mm and height 20 mm were prepared for the heat treatment. Heat treatment conditions of the samples are shown in Table 2. In order to capture photograph only at a given point of the sample, relatively deep grooves on the

surface of the samples were created and photography was done by the end of the groove for each sample. After preparing 11 samples, section them and create three-dimensional microstructure, Rockwell C hardness test was performed to compare the morphology and geometric properties of the microstructure with hardness and mechanical properties of the samples. The test apparatus was Controlab Super Rockwell-Duplex-713-SR.

2.3 *Image Analysis Method*

Images that are often used in the image processing were taken by digital cameras and grayscale were being treated. Algorithms used in the image processing techniques are capable of identifying all phases and separating them in the image. However, these algorithms are also associated with the problems that are fully described in the reference [19]. In the present study, there is an effort to use various techniques such as gray scaling and binarizing of photos to maximize the identification efficiency. Figure 4 shows the steps of converting optical microscope image to grayscaled and binarized one that is ready for the image analysis.

To study the geometric properties of two-dimensional images at any section of the sample, the image-processing program that was coded in Visual Basic programming language by authors, has been employed and the 3D-Doctor commercial image analysis software was used for the reconstruction of 3D microstructures.

Particle-based method for geometrical properties calculation has been used in this research. According to this method, each particle can be easily categorized and segregated. Geometric properties include shape parameters and location relative to other particles at each sample is calculated for each single particle. Properties needed to evaluate the shape; size and distribution of particles are area, perimeter, and Feret diameter. The area of each particle was calculated by counting the pixels belonging to the particle and multiplying by the image magnification factor. Perimeter of each particle is calculated according to the following formula:

$$P = \frac{4}{\pi} (\text{Pro}0^\circ + \text{Pro}45^\circ + \text{Pro}90^\circ + \text{Pro}135^\circ) \quad (1)$$

Where Pro is the length of the image projection in the direction of 0°, 45°, 90° and 135° (see Figure 5 [20]).

Feret diameters of angles from 0 to 175 degrees with 5 degrees interval are calculated, and the largest Feret diameter of the particle is considered being an estimate of the diameter. In this research, particle-based parameters will be used for the classification of chromium carbides.

Volume percent and specific surface area of the particles can be calculated due to the stereological equations from 2D image of the microstructures [21]. Delesse [22], Rosiwal [23], and Thompson [24], proved that surface, linear and point density as surface, linear and point analysis can be used to calculate volume density (see Table 3). Specific surface area can be calculated only by performing linear and surface analysis. In 1945 Saltykov [25] published the proof of this formula:

$$S_V = \frac{4}{\pi} L_A = 2N_L \quad (2)$$

Where L_A is the length of lines per unit area and N_L is the number of points per unit length.

The equation to calculate the volume density can be applied to all homogenous structures, even if the microstructure is not isotropic. Anisotropic two-dimensional structures can act inconsistently [17]. In this case, a three-dimensional image of the desired structure is needed. Three-dimensional analysis is inevitable when the microstructure doesn't have structural symmetry or convex and recurring shape. Such a structure, depending on the shape and measurements of the geometrical parameters, could look very different in two and three dimensions.

3. Result and Discussion

3.1 *Two-dimensional Analysis of Carbide Morphology*

To study the two-dimensional microstructure of carbides in the tool steel, area density and maximum Feret diameter, is calculated for each sample and shown in Table 4. Due to the Table 2, the heat treatment conditions of the samples can be divided into two general categories:

1. Effect of austenitizing temperature: This category is divided into the following four groups that these subclasses in the quenching media, tempering temperature and number of tempering are fixed and only the austenitizing temperature is variable. These subgroups are defined as follows:
 - Subgroup 1-1: Samples 2 and 3.
 - Subgroup 1-2: Samples 5 and 6.
 - Subgroup 1-3: Samples 7 and 9.
 - Subgroup 1-4: Samples 10 and 11.
2. The effect of tempering temperature: This category is divided into the following three groups that these subclasses in the quenching media, austenitizing temperature and number of tempering are fixed and only the tempering temperature is variable. These subgroups are defined as follows:
 - Subgroup 2-1: Samples 1, 4 and 8.
 - Subgroup 2-2: Samples 2, 5 and 7.
 - Subgroup 2-3: Samples 3, 6 and 9.

Certainly , any of the parameters presented above, which include the austenitizing and tempering temperature , have a direct impact on the volume density, size and morphology of carbides in tool steel. Further, analyzing each of these parameters by using 2D and 3D methods will be performed, and metallurgical reasons will be discussed.

3.1.1 *Effect of Austenitizing Temperature*

When the austenitizing temperature is higher, this leads to the further dissolution of carbides and carbon enriched austenite. After quenching this carbon, enriched austenite transforms to the high-carbon martensite and relatively large amount of retained austenite because of dramatically decrease in the martensite start temperature. So it can be expected that this high carbon retained austenite during tempering is susceptible to more and smaller carbide precipitation because of more uniform distribution of carbon and other alloying elements. In Table 5, carbide, retained austenite and martensite density in AISI D2 tool steel at various austenitizing temperatures after quenching in oil is shown from reference [26]. According to sub-groups 1-1, 1-2, 1-3 and 1-4 and Table 4, Figures 6 and 7 show the variation of density and the size of the carbide phase respectively for the samples included in these subgroups. In Figure 6, increasing can be seen in the carbide phase density in subgroups of 1-1 and 1-3 by increasing in austenitizing temperature from 980 to 1020°C, due to the higher carbide dissolution in 1020°C. Figure 7 also shows that these further dissolution and distribution lead to nucleation and growth of smaller size carbides. But in the subgroup 1-2 it can be seen that the difference of the carbide phase density of the two austenitized sample in 980°C and 1020°C is very impressive and more than other subgroups. This significant difference can be studied due to the tempering temperature of this subgroup. According to reference [27] and Figure 8 taken from reference [28] for AISI A2 tool steel with chemical composition: 1%wt C, 5.20%wt Cr, 1%wt Mo and 0.25%wt V, can be found that in high chromium cold work tool steels in tempering temperature range from 250 to 400°C, decomposition of retained austenite can lead to dramatically increase in amount of carbide precipitation. Therefore, the difference in the amount of carbides in subgroup 1-2 can be investigated because of the complex effects of high amount of retained austenite after quenching and higher dissolution of carbon in austenite during austenitizing. These two factors interact with each other because of carbon enriched austenite lead to decrease in martensite start temperature and thereby increase the amount of retained austenite. In Figure 7, in subgroups 1-2 and 1-4, an opposite procedure can be seen in the calculated Feret diameter compare to subgroups 1-1 and 1-3.

This opposite procedure may be because of tempering temperature of these subgroups that lead to the abnormal growth of carbides during tempering.

In order to verify the calculated values for density and Feret diameter of carbides, and comparison of the different samples defined in this section, the images of two-dimensional metallographic of samples included in subgroups of 1-1, 1-2, 1-3 and 1-4 are shown in Figure 9.

3.1.2 Effect of Tempering Temperature

In Figure 10 , [29] it can be found that in AISI D2 tool steel, tempering and austenitizing temperature both and alone have a direct impact on its hardness. As can be seen in Figure 10, when the tempering temperature increases, the hardness drops, but when the austenitizing temperature of steel is 1070°C, after quenching, retained austenite is more and hardness is less than austenitizing temperature of 1030°C. Because of more dissolution of carbide and enriched martensite phase in 1070°C austenitizing temperature, after quenching, secondary hardening and secondary carbide phases is more when the steel tempered in 530°C.

According to subgroups 2-1, 2-2 and 2-3, and their heat treatment conditions, hardness of samples include in these subgroups are shown in Table 6.

According to Table 6, Figure 11 plotted for the better comparison of samples' hardness. In Figure 11, it can be seen that when the tempering temperature increases up to 530 °C, hardness drops but, at 530°C, secondary hardening may lead to increase in the hardness. Figures 12 and 13 show the variation of density and the size of the carbide phase, respectively for the samples included in 2-1, 2-2 and 2-3 subgroups. In the samples included in the subgroup 2-1 (samples 1, 4 and 8) after quenching in the air and cooled in the equilibrium condition from austenitizing temperature and due to the continuous cooling transformation diagram (CCT) of AISI D2 tool steel [24] that have been shown in Figure 14, can be found that more contributions of carbide precipitation occurs during cooling from the austenitizing temperature. When the tempering temperature rises from 180°C in sample 1 to 530°C in sample 8, carbide phase density

decreases as shown in Figure 12. That could be because of the higher dissolution rate of non-alloy carbides (Epsilon and Eta) compared with secondary carbides precipitation. Likewise, dissolution of primary and precipitation of fine secondary carbides as shown in Figure 13, leads to reducing the overall size of carbides. As shown in Figure 12, carbide phase density in the sample 3 is less than sample 9. This difference, because of the sample 9 quenched in the air, has more opportunity to carbide precipitation. The samples included in the subgroup 2-2, because of quenching in salt bath and don't have any opportunity for carbide precipitation during cooling from the austenitizing temperature, so they are more susceptible to carbide precipitation during tempering. This effect is well visible in samples 2 and 5. When the tempering temperature is 530°C, due to the relatively high rate of dissolution of primary carbides in sample 7, compared with secondary alloyed carbides precipitation, the overall density of carbides is reduced. But as mentioned before, due to the fine secondary carbides precipitation, carbides average size decreases.

In samples included in subgroup 2-3, because of low austenitizing temperature (980°C), secondary hardening and secondary carbide formation have not been observed but because of the increase of tempering temperature, hardness has dropped and the density of carbide phase raised due to martensite transformation to the primary carbides and because of the abnormal growth of primary carbides, average size has increased dramatically.

In order to verify the calculated values for the density and Feret diameters of carbides, and comparison between the different samples in the subgroups defined in this section, the images of two-dimensional metallographic of samples included in subgroups 2-1, 2-2 and 2-3 are shown in Figure 15.

3.2 *Three-dimensional Analysis of Carbide Morphology*

By using three-dimensional analysis, the morphological parameters of the microstructure can be calculated that by the two-dimensional analysis is impossible, such as Gaussian curvature and the mean curvature that can be achieved only by three-dimensional imaging. Another advantage of the three-

dimensional analysis is that the microstructure can be analyzed directly, without any pre-assumptions about the shape or size of the particles.

To study the morphology of the carbides using three-dimensional analysis, the field parameters of volume and surface are calculated and shown in Table 7. These field parameters can be used as a tool to investigate the three-dimensional morphology of microstructures. In Table 7, V represents the volume and S represents the surface of carbide phase.

In order to verify the stereological equations (see Table 3), the mean volume and area density of carbide phase in all sections, for all samples are calculated, and shown in Table 8. According to Table 8, it may be perceived that stereological equations presented in Table 3 are held with reasonable tolerances.

3.2.1 Describe the Three-dimensional Microstructure

By combination of the field parameters introduced in the previous section, a number of other geometric parameters are defined to describe the three-dimensional microstructure of carbide phase. The three-dimensional microstructure factors, f_1 and f_2 are defined as follows:

$$f_1 = 6\sqrt{\pi} \frac{V}{\sqrt{S^3}} \quad (3)$$

$$f_2 = \frac{6V}{\pi d_{max}^3} \quad (4)$$

Values of the above mentioned parameters are between 0 and 1 and carbides shape compared with spheres and its deviation from a sphere is calculated. Geometric parameters f_1 compare the volume of carbide with volume of a sphere with equal surface. In equation 2, d_{max} is the three-dimensional maximum Feret diameter. Three-dimensional geometrical parameters (f_1 and f_2) are calculated for all samples and presented in Table 9.

As seen in the preceding sections that analysis of the shape of carbides performed in two dimensions, and metallurgical aspects of the heat treatment was evaluated according to the two-dimensional geometrical

parameters, analyses can be evaluated using a three-dimensional analysis by calculation of volume density, three-dimensional average carbide size, and three-dimensional geometric parameters. Due to Table 9 and Table 4, it may be perceived that two-dimensional and three-dimensional analysis of microstructure can often be done in the same manner.

In Table 10, the average carbide size for each sample was calculated by three-dimensional and two-dimensional analysis. Due to Table 10, it can be found that matching of the calculated values for carbides in both two-dimensional and three-dimensional analysis is acceptable.

In continue, the reconstructed three-dimensional microstructures of all samples examined in the present study, are shown in Figure 16. As seen in the microstructures shown in Figure 16, it may be perceived that the austenitizing and tempering temperature have a direct impact on the morphology, size and continuity of the carbide phase.

4. Conclusion

In this study, it can be found that when austenitizing temperature rise rises, this leads to further dissolution of carbide in austenite and after quenching and tempering, alloy carbides density raises due to the dissolution of alloying elements during austenitizing and precipitate small secondary carbides in the tempering stage. Increasing the tempering temperature from 180°C to 530°C causes the secondary carbides precipitation and consequently secondary hardening. The size of the secondary carbides is much smaller than the primary carbides. The results from the two-dimensional and three-dimensional analysis have some differences in the exact values but similar trends are indicated to evaluate the metallurgical aspects.

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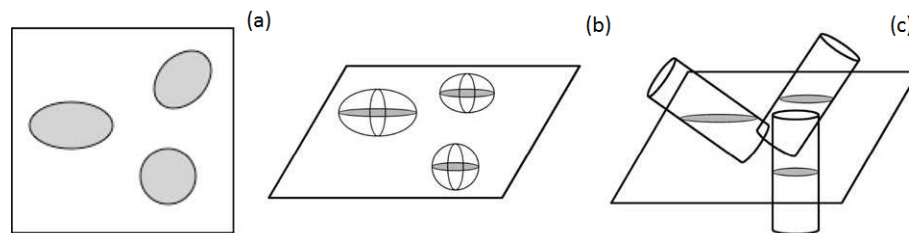


Figure 1

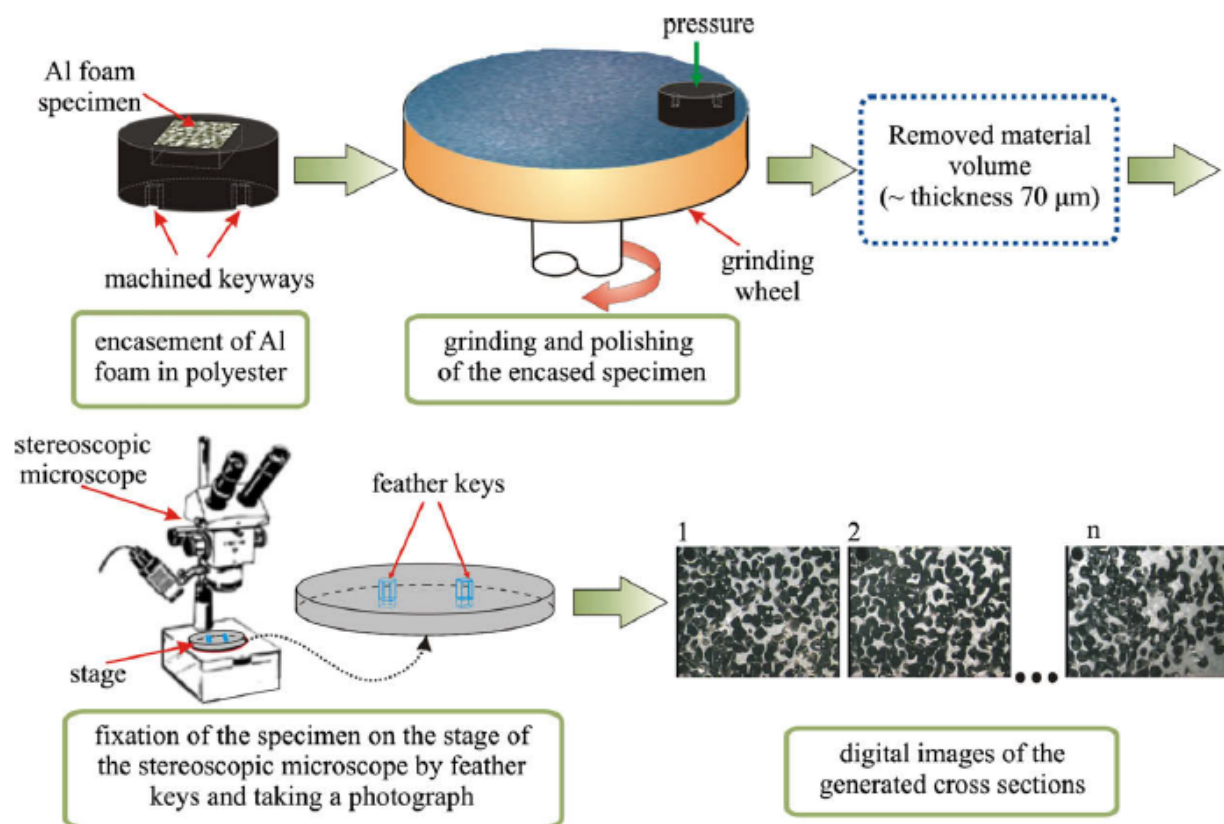


Figure 2

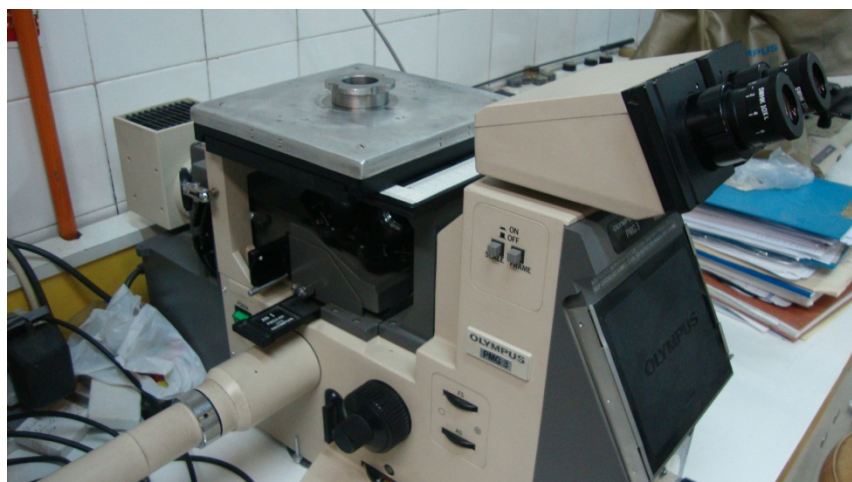


Figure 3

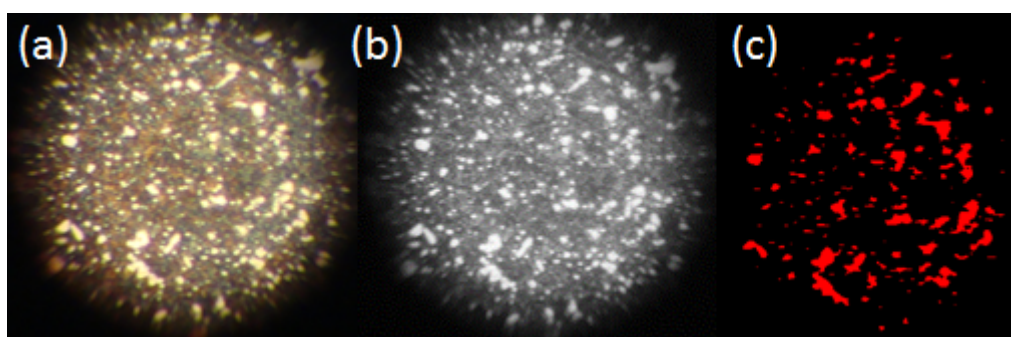


Figure 4

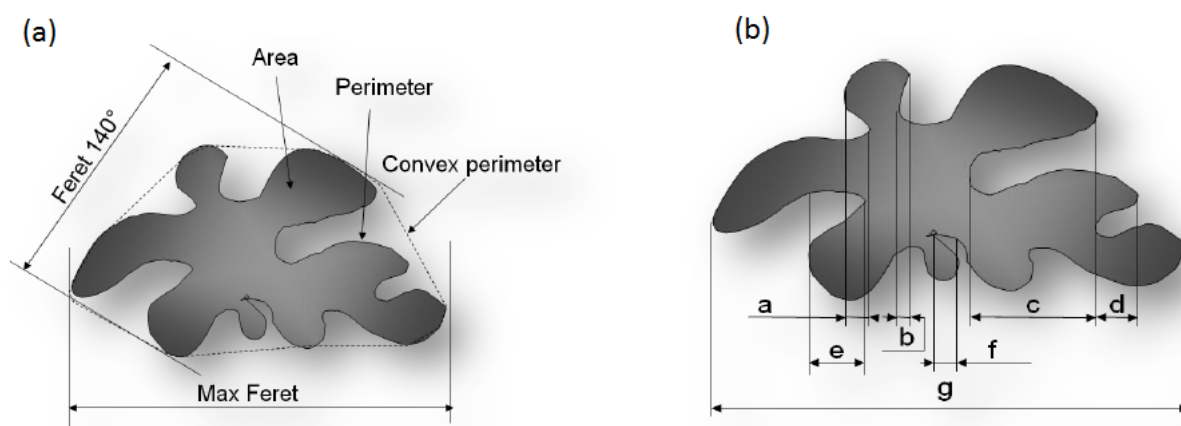


Figure 5

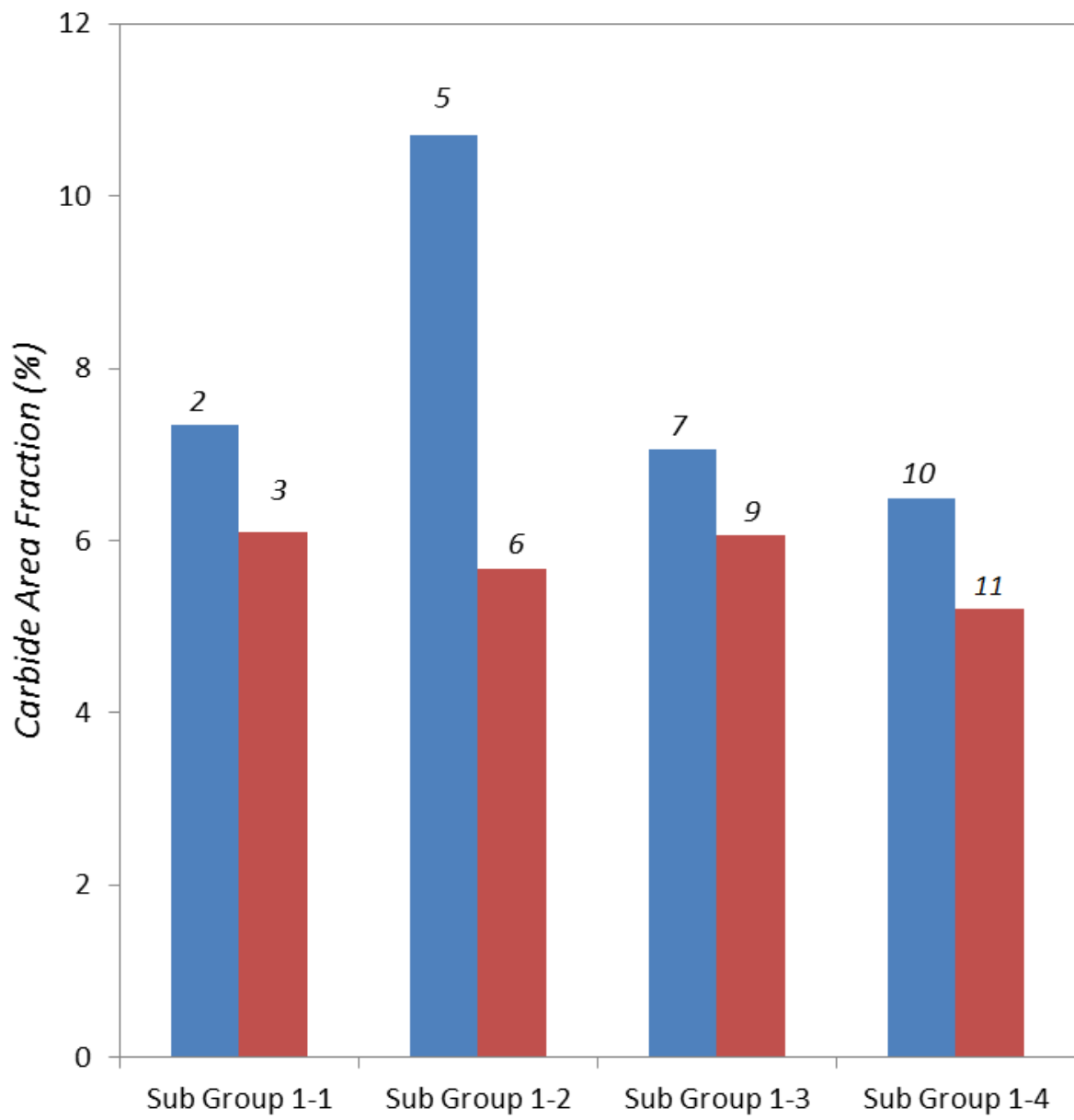


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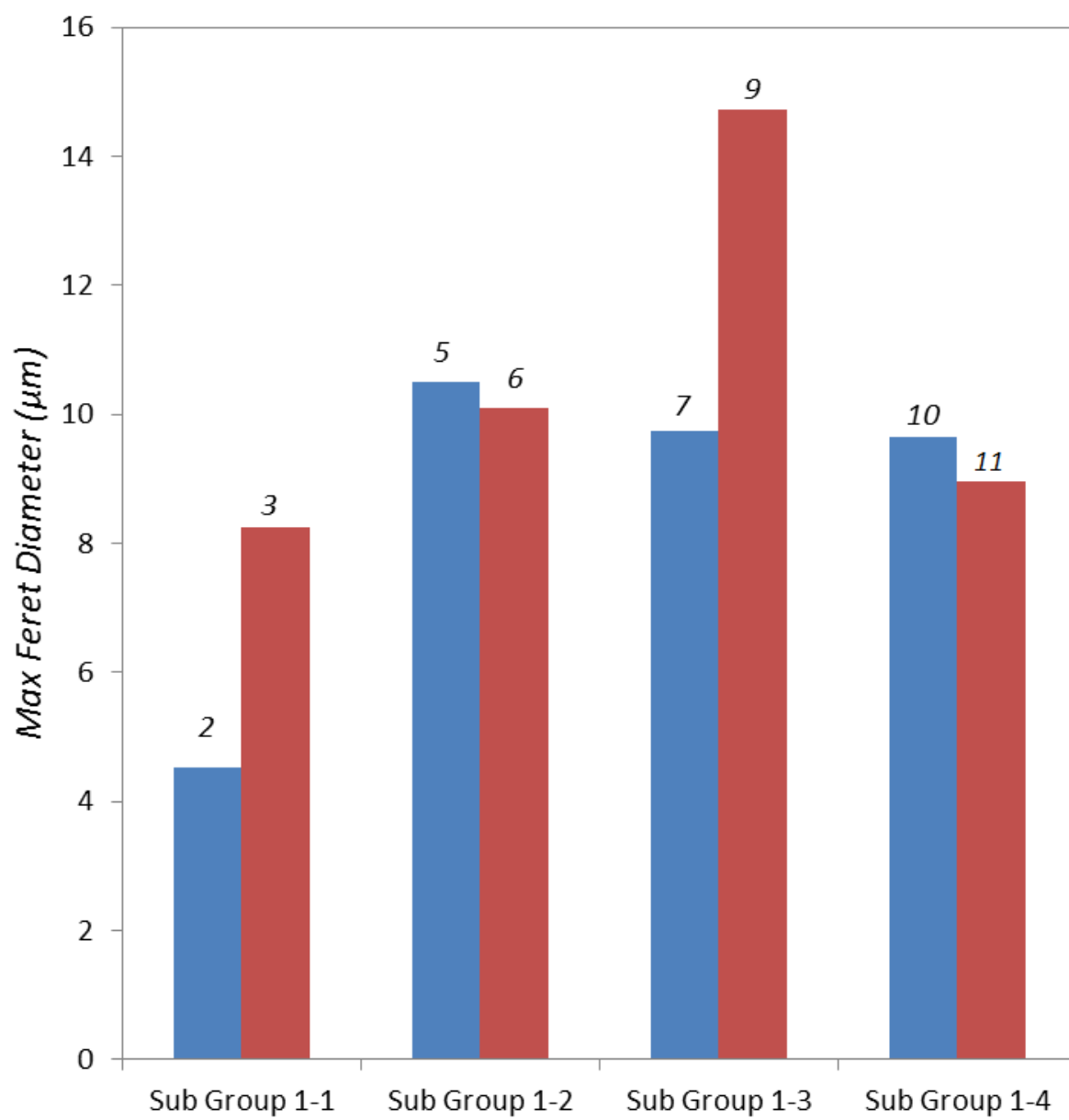


Figure 7

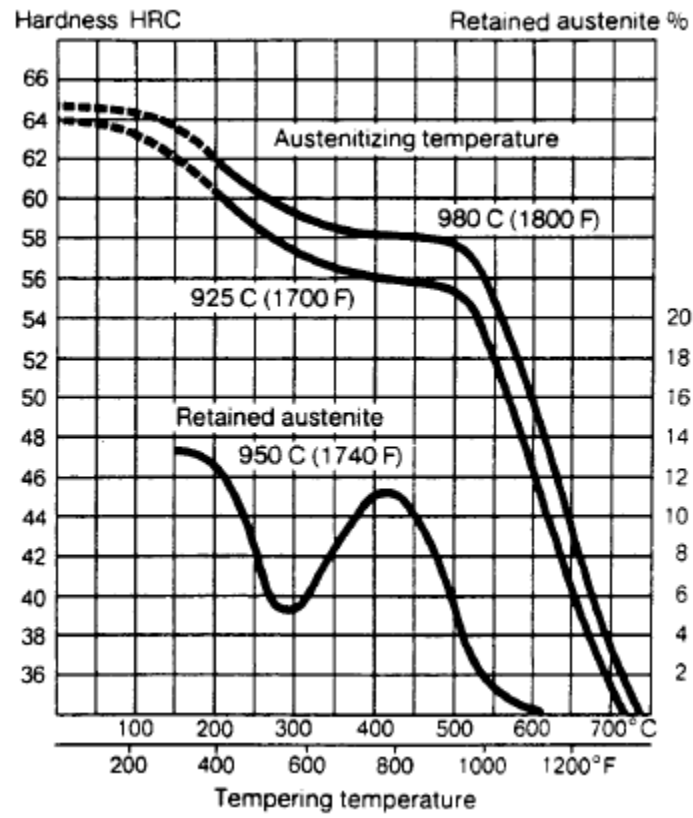


Figure 8

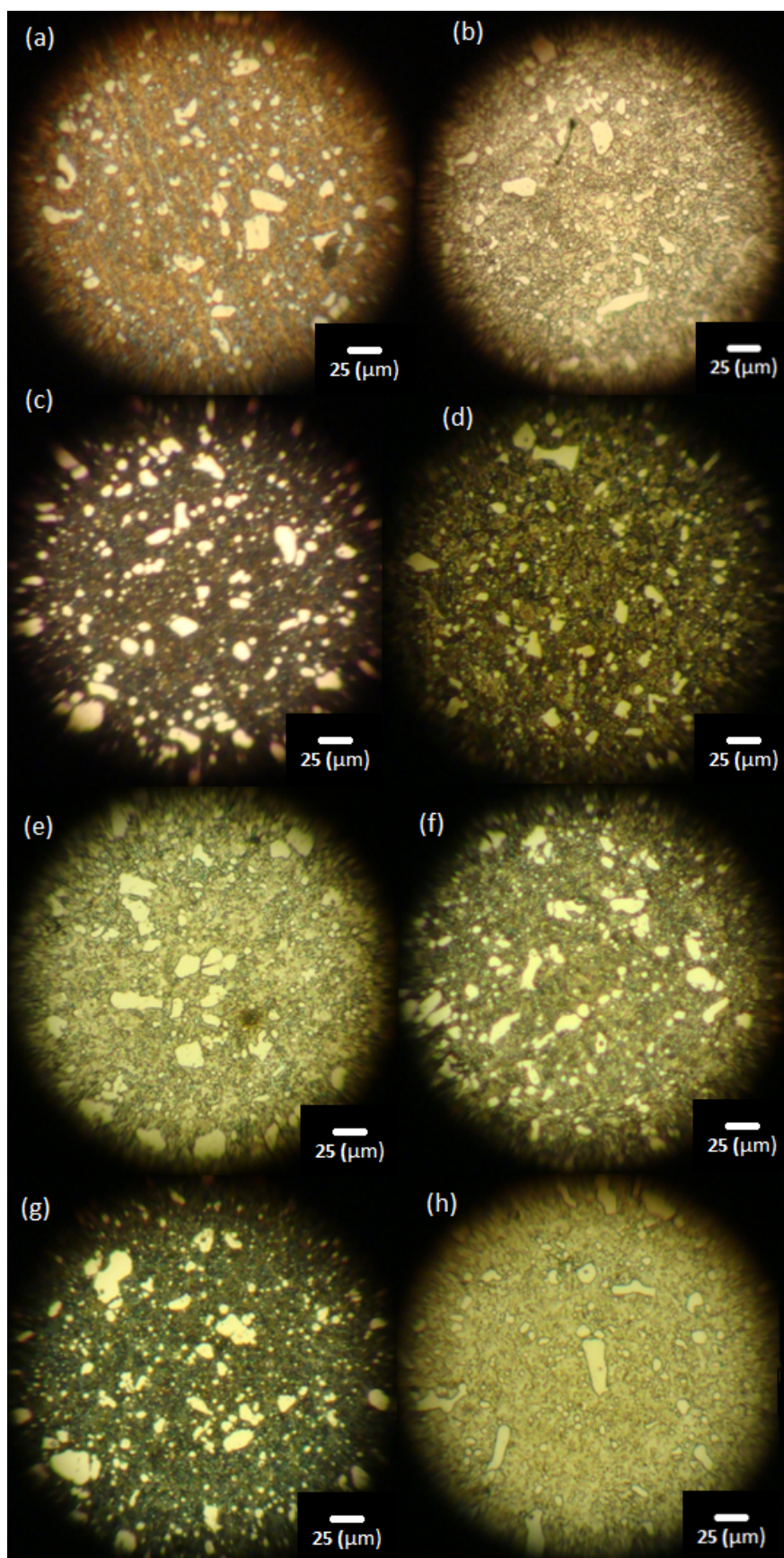


Figure 9

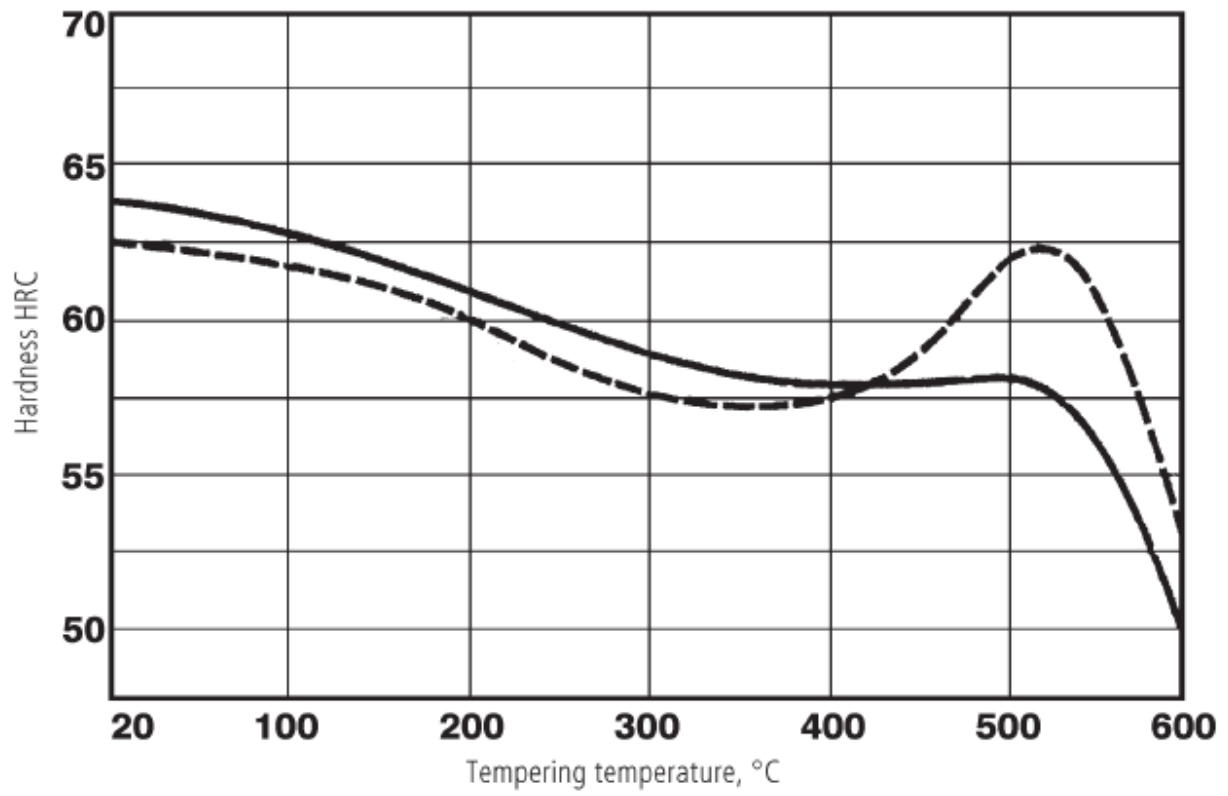


Figure 10

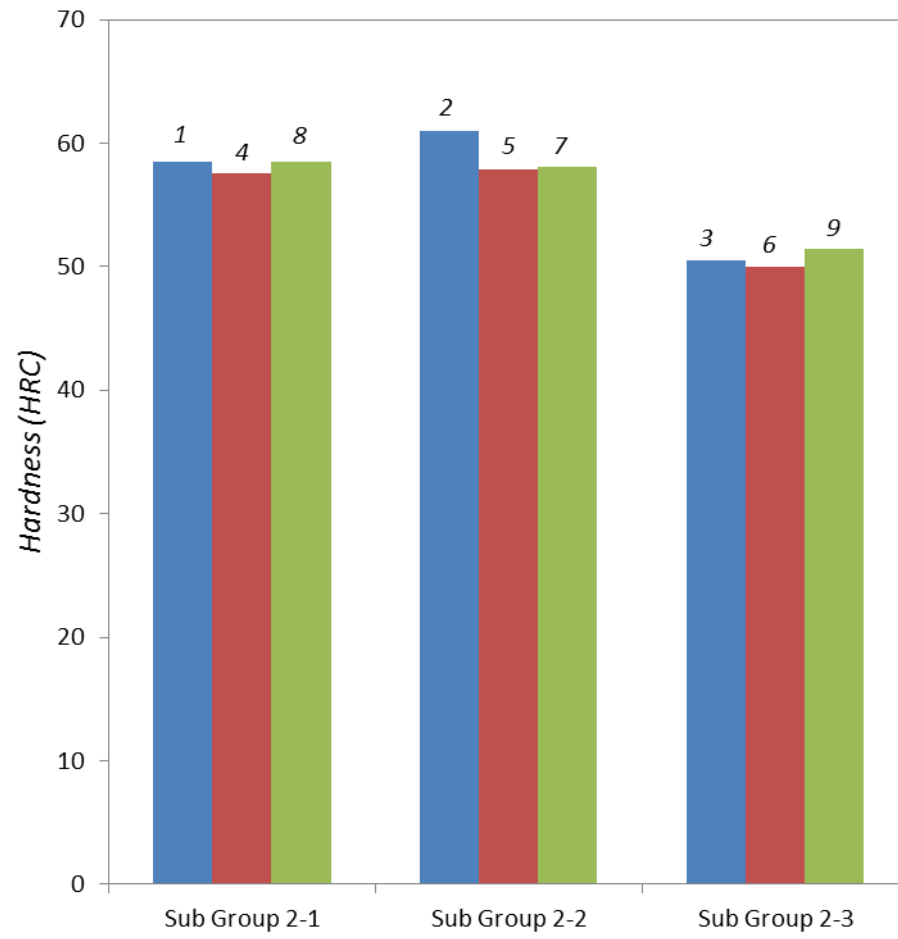


Figure 11

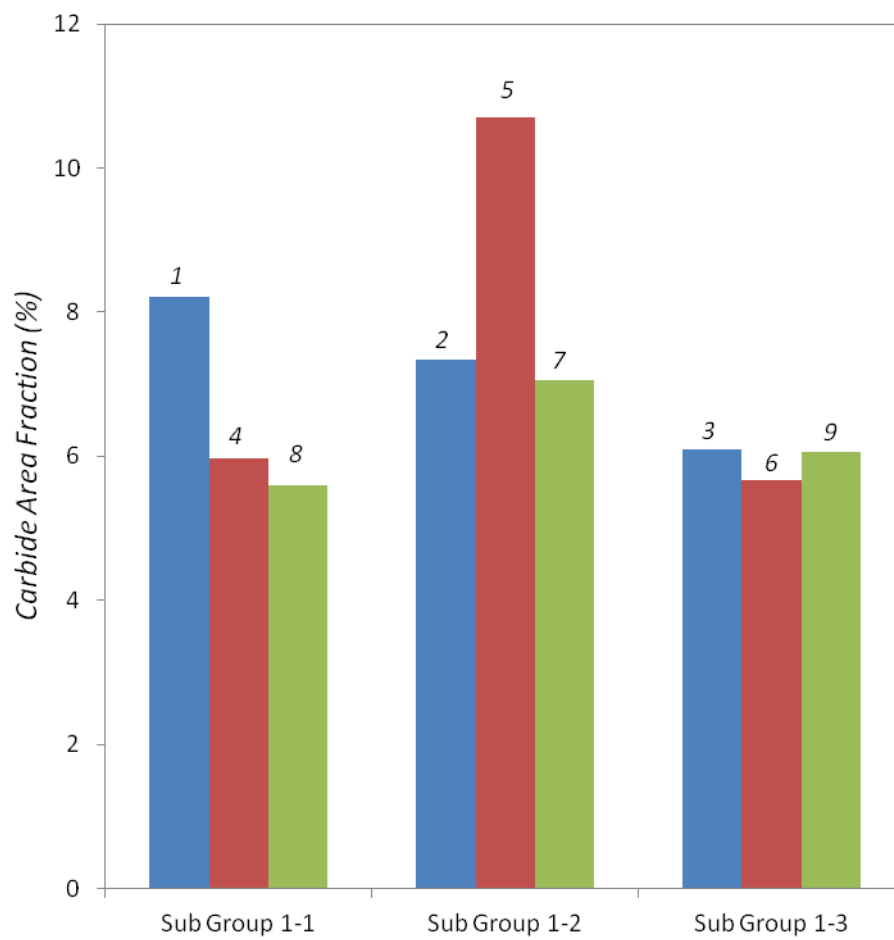


Figure 12

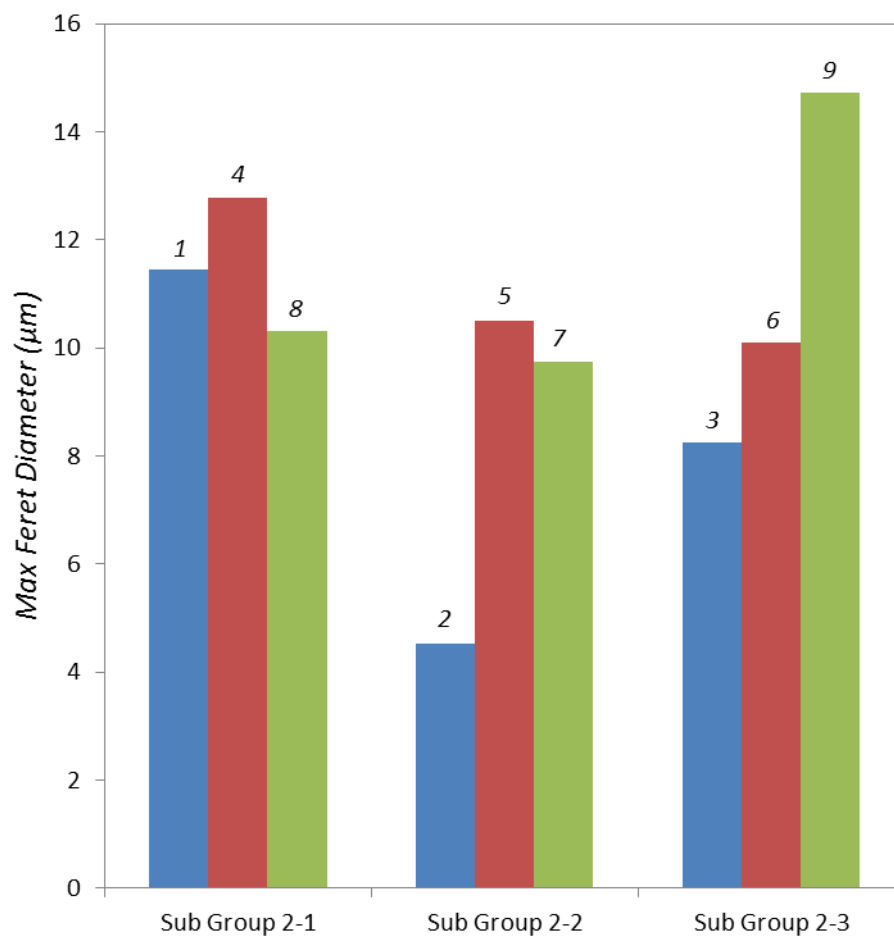


Figure 13

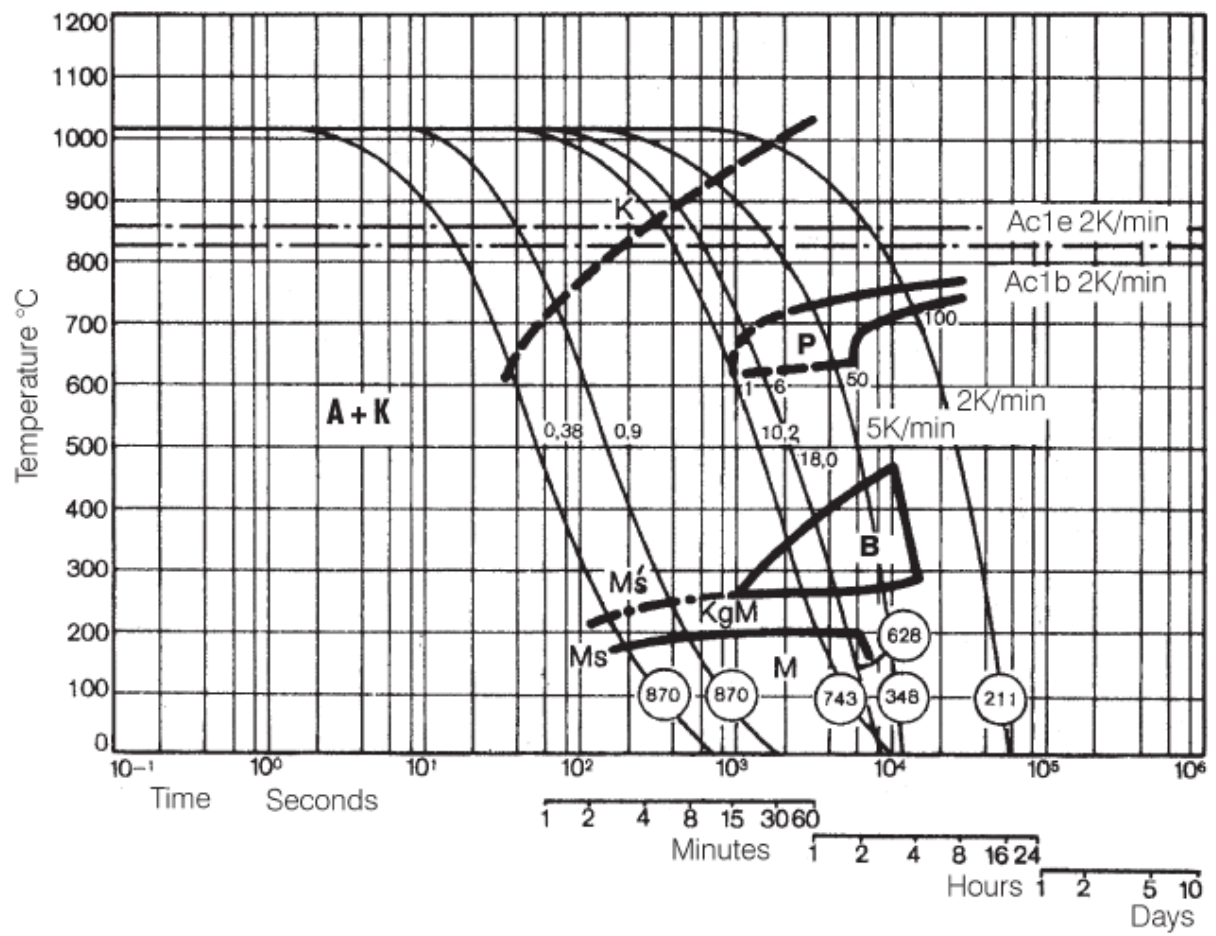


Figure 14

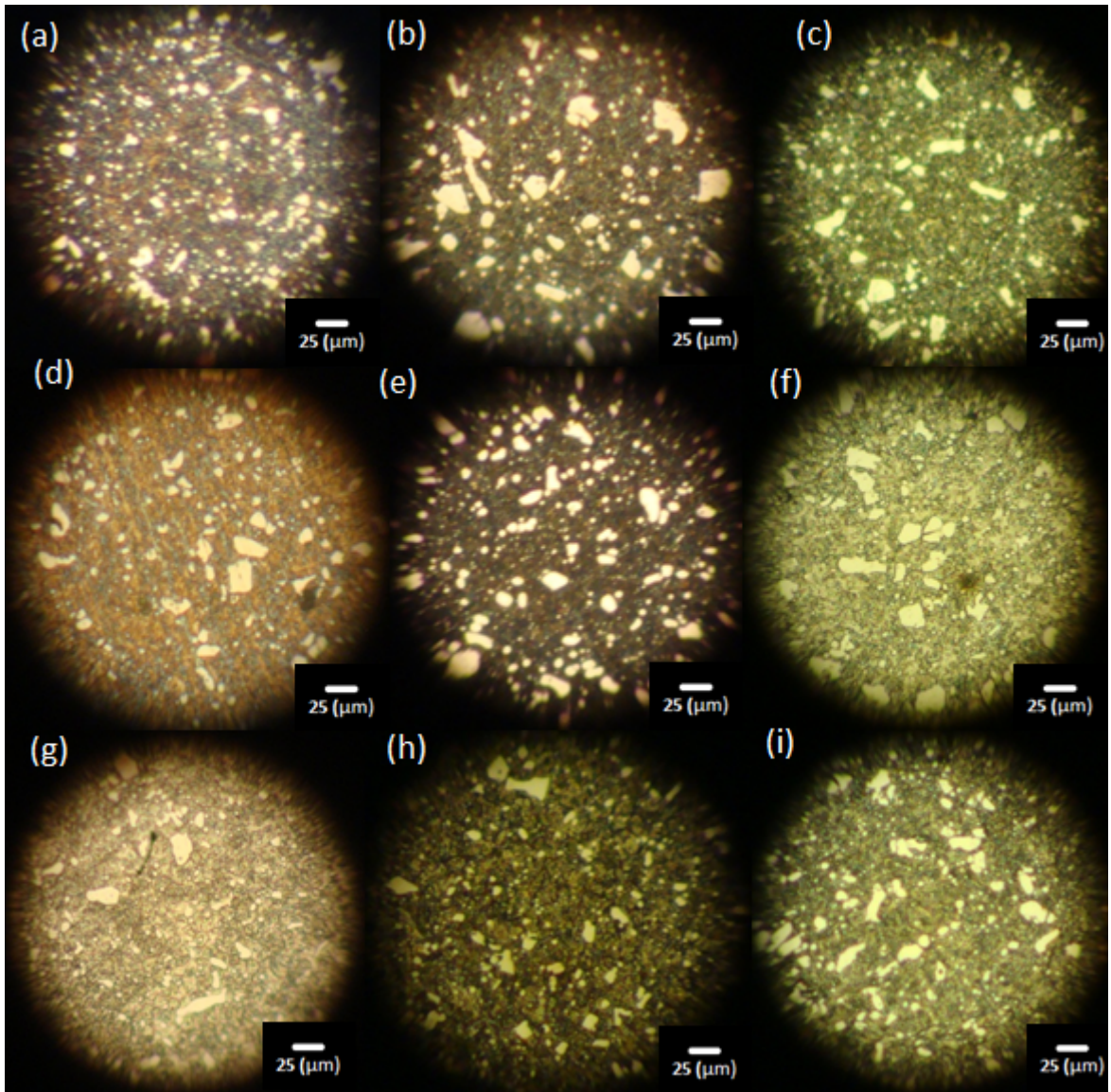


Figure 15

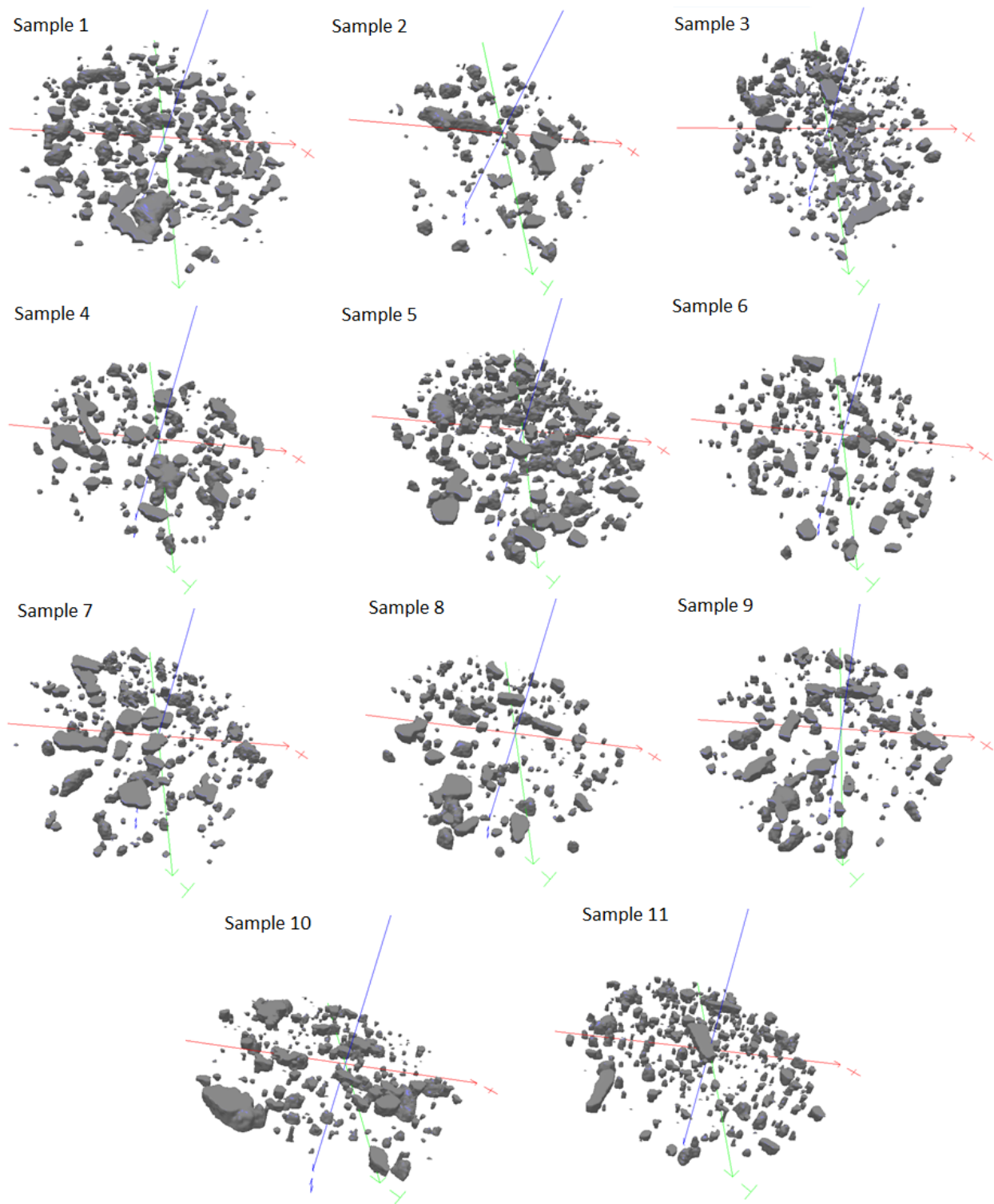


Figure 16

Table 1

Chemical Composition	C	Si	Mn	Cr	Mo	V	Fe
%Average wt	1.55	0.30	0.30	11.30	0.75	0.75	Balance

Table 2

Sample No	(°C) Austenitizing Temperature	Quench Media	(°C) Tempering Temperature	Number of Tempering
1	1020	Air	180	1
2	1020	Salt Bath	180	1
3	980	Salt Bath	180	1
4	1020	Air	400	1
5	1020	Salt Bath	400	1
6	980	Salt Bath	400	1
7	1020	Salt Bath	530	1
8	1020	Air	530	1
9	980	Salt Bath	530	1
10	1020	Salt Bath	400	2
11	980	Salt Bath	400	2

Table 3

Spatial Parameter	Area Method	Linear Method	Point Method
V_V	$A_A =$	$L_L =$	$P_P =$
S_V	$\frac{4}{\pi} L_A =$	$2N_L =$	
M_V	$2\pi\chi_A =$		
K_V			

Table 4

Sample No	1	2	3	4	5	6	7	8	9	10	11
(%) Area Density of Carbide Phase	8.22	7.34	6.10	5.97	10.71	5.67	7.05	5.60	6.06	6.49	5.21
Maximum Feret Diameter (μm)	11.44	4.52	8.26	12.78	10.49	10.09	9.75	10.32	14.73	9.64	8.97

Table 5

Austenitizing Temperature (°C)	(% Volume)		
	Martensite	Retained Austenite	Carbide
1040	79	7	14
1065	65	22	13
1095	33	55	12
1120	5	85	10
1135	2	88	10
1150	2	88	10

Table 6

.Sample No	1	2	3	4	5	6	7	8	9
Hardness (HRC)	58.5	61	50.5	57.6	57.9	50	58.1	58.5	51.4

Table 7

.Sample No	Carbide Volume	Carbide Surface
	V, μm^3	S, μm^2
1	259382	143290
2	224946	62681
3	186820	120153
4	182827	96854
5	328144	173752
6	173766	85566
7	215912	112276
8	171621	81107
9	175726	101521
10	198828	103484
11	159809	109571

Table 8

.Sample No	Carbide Volume Fraction	Carbide Area Fraction
	$V_{\text{Carbide}}/V_{\text{Total}}\%$	$A_{\text{Carbide}}/A_{\text{Total}}\%$
1	8.24	8.22
2	7.15	7.34
3	5.94	6.10
4	5.81	5.97
5	10.43	10.71
6	5.52	5.67
7	6.86	7.05
8	5.45	5.60
9	5.90	6.06
10	6.32	6.49
11	5.08	5.21

Table 9

.Sample No	f_1	f_2
1	0.051	0.333
2	0.1524	0.310
3	0.0477	0.402
4	0.0645	0.322
5	0.0482	0.309
6	0.0738	0.322
7	0.0610	0.406
8	0.0790	0.313
9	0.0610	0.340
10	0.0635	0.370
11	0.0468	0.318

Table 10

.Sample No	1	2	3	4	5	6	7	8	9	10	11
<i>Max Feret Diameter (μm 2-Dimensional)</i>	11.44	4.52	8.26	12.78	10.49	10.09	9.75	10.32	14.73	9.64	8.97
<i>Max Feret Diameter (μm 3-Dimensional)</i>	8.05	6.84	7.66	7.56	7.77	7.74	7.54	8.39	8.08	7.05	8.05

Figure Captions:

Fig 1.: One of the main problems in the two-dimensional microstructure analysis, two-dimensional microstructure of (a) can be somewhat elliptical shape (b), non-parallel cylindrical shape (c) or any other form of three-dimensional shape [2].

Fig 2.: Scheme of continuous exfoliation and photographed with optical microscopy was performed to examine the three-dimensional structure of aluminum foam [13].

Fig 3.: View from the flange and sheet made of aluminum to hold the sample and preventing rotation.

Fig 4.: Metallographic image processing, (a) optical microscope image of sample, (b) Grayscale photo developed by image processing software, (c) binarized image created by software which is intended only carbide phase.

Fig 5.: (a) The geometric parameters of a particle, (b) an example of the image projection along the 90° : $\text{Pro}90^\circ = \mathbf{a} + \mathbf{b} + \mathbf{c} + \mathbf{d} + \mathbf{e} + \mathbf{f} + \mathbf{g}$ [15].

Fig 6.: Area fraction of the carbide phase for the following subgroups: 1-1, 1-2, 1-3 and 1-4.

Fig 7.: The mean maximum Feret diameter of carbide phase for subgroups 1-1, 1-2, 1-3 and 1-4.

Fig 8.: Hardness and the amount of retained austenite as a function of tempering temperature for tool steel AISI A2 [23].

Fig 9.: Metallographic images of samples in the subgroups 1-1, 1-2, 1-3 and 1-4, (a) sample 2, (b) sample 3, (c) sample 5, (d) sample 6, (e) sample 7, (f) sample 9, (g) sample 10 and (h) sample 11.

Fig 10.: Effect of austenitizing temperature and tempering temperature on the hardness of steel AISI D2 after tempering, broken line is austenitizing temperature of 1070°C and the solid line austenitizing temperature of 1030°C [24].

Fig 11.: Hardness of samples in subgroups 2-1, 2-2 and 2-3.

Fig 12.: Area fraction of the carbide phase for the following subgroups 2-1, 2-2, 2-3.

Fig 13.: The mean maximum Feret diameter of carbide phase for subgroups 2-1, 2-2, 2-3.

Fig 14.: Continuous cooling transformation diagram (CCT) of cold work tool steel AISI D2 [24].

Fig 15.: Two-dimensional metallographic images of samples in subgroups 2-1, 2-2 and 2-3, (a) sample 1, (b) sample 4, (c) sample 8, (d) sample 2, (e) sample 5, (f) sample 7, (g) sample 3, (h) sample 6 and (i) sample 9.

Fig 16.: Three-dimensional microstructure of all samples.

Tables Captions:

Table 1: The chemical compositions of AISI D2 tool steel.

Table 2: Heat treatment conditions of samples.

Table 3: Stereological equations; V_V is volume density, S_V is surface to volume ratio, M_V is mean curvature to volume ratio and K_V is Gaussian curvature to volume ratio of secondary phase in three-dimensional analysis; A_A is area density, L_A is line length to area ratio and χ_A is Euler number to area ratio of secondary phase in two-dimensional analysis; L_L is line length density and N_L is number of point to line length ratio of secondary phase in one-dimensional analysis; P_P is point density of secondary phase in point analysis method.

Table 4: Results of two-dimensional metallographic analysis (tolerances for maximum Feret diameter is $\pm 1.5 \mu\text{m}$).

Table 5: Amount of different microstructures in AISI D2 tool steel versus austenitizing temperature [21].

Table 6: Hardness of the sample in the subgroups 2-1, 2-2 and 2-3.

Table 7: Three-dimensional parameters of samples (overall volume is $381 \times 413 \times 20 \mu\text{m}^3$).

Table 8: Carbide volume fraction and carbide area fraction of each sample (overall volume is $381 \times 413 \times 20 \mu\text{m}^3$).

Table 9: Three-dimensional microstructures factors for each sample.

Table 10: The average carbide size in two and three dimensional analysis for all samples.