HEAT TREATMENT INFLUENCE ON CARBIDIC PHASE DISTRIBUTION AND HARDNESS OF PM HSS WITH NIOBIUM ADDITION

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Abstract
Powder metallurgy allows the preparing of such materials that are not possible to prepare by conventional melt metallurgy. Among these materials also belongs the high speed steel M2 (STN 41 9830), which was alloyed by 1.9% Nb. By suitable selected parameters of heat treatment we tried to achieve not only homogeneous distribution of carbide phases, but also shape uniformity. Digital image analysis and stereological methods were used for evaluation of these characteristics. It was determined that the proposed heat treatment is suitable, because distribution and size of carbidic phases is isotropic and the shape of carbidic phases is homogeneous. The hardness of experimental material after optimal heat treatment was 65 HRC.
Keywords: PM high speed steel, carbidic phase, heat treatment, image analysis, size and shape characteristics, planar distribution, Voronoi tessellation, hardness

INTRODUCTION

Tool steels prepared by powder metallurgy are the object of intensive research in all advanced countries in the world. The reason is that PM is able to produce a complex shape of structural parts from material with properly modified chemical compositions. This production is low cost, energy saving and ecologically friendly [1-3].

On the basis of knowledge concerning the influence of individual carbide forming elements it carried over to the range of steels in which a favourable effect of vanadium and cobalt is utilised, and in this manner the content of tungsten and molybdenum is lowered. In high-speed steels it is important that their basic substance resists against tempering at high temperatures and the hardest carbides were distributed in matrix in such as manner that they will increase the wear resistance at full blast. High-speed steels contain, besides tungsten, also chromium for an increase of oxidation resistance and for a hardness increase, molybdenum and vanadium for carbide formation and resistance against tempering at red heat [4]. Cobalt is added for a hardness increase at high temperatures, it increases secondary hardness but thereby toughness and bending strength is lowered. It increases the transformation temperature $\alpha \rightarrow \gamma$, and in this way a resistance of solid solution α against the loss of hardness during heating is increased. Cobalt, at the same time, increases a measure of transition of W and Mo from martensite to carbides during tempering by lowering the solubility in α solid solution. By this mechanism cobalt supports precipitation strengthening, increases secondary hardness, improves thermal conductivity, but worsens the toughness [4-7]. By niobium addition there is achieved formation of stabile primary carbides NbC that act as an inoculant. Grain refinement and improvement of carbide distribution it also attained. Niobium prevents a grain coarsening during heat treatment but

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its content is limited to 1.5%. In the case of higher content, the primary niobium carbides are coarsened, leading to deterioration of material properties [1,8].

The aim of this contribution is knowledge of relations between the microstructure and heat treatment of PM high-speed steel prepared by rapid solidification and alloyed by niobium. Tool steels prepared by this technology are characterised by optimal isotropic microstructure with the presence of high dispersive and uniformly distributed carbidic phases, a carrier of high hardness. For this reason it is important a distribution of these phases be correctly statistically evaluated and quantified. Carbidic phases were defined by light microscopy and processed by image analyser. The size, shape and planar distribution of carbidic phases were quantified by selected geometric characteristics using stereological methods.

EXPERIMENTAL MATERIALS AND METHODS

Experiments were carried out on PM high speed steel M2 (STN 41 9830) fine alloyed with Nb (1.3C, 6.5W, 4.3Cr, 1.9V, 5Mo, 1.9Nb) [9,10], produced by a rapid solidification by melt spraying into nitrogen using the apparatus of fy Osprey. Powder material was compacted by hot isostatic pressing (HIP) and then subjected to heat treatment (HT) [10,11]. We selected heat treatment published in [12], austenitizing at 1100°C/20 min in a vacuum with subsequent hardening at pressure 0.5 MPa in a nitrogen medium; tempering at 540°C/3x1h, marked **G**. The comparative heat treatment was carried out according to [11,13], austenitizing at 1190°C/15min in a vacuum with subsequent hardening at 5bar in a nitrogen medium; tempering at 560°C/1h, 540°C/1h, 520°C/1h, marked **B**. A polished metallographic cut was etched for carbide visualisation by 4% NaOH in a saturated solution of $KMnO_4$. The samples were observed by a light microscope OLYMPUS GX71 with digital camera DP71 allowing image acquisition with 4080 x 3072 pixels of resolution. DIPS 5.0 [14] and ImageJ [15] were used for image processing and analysis.

RESULTS

Microstructure

Porosity of the material after hot isostatic pressing and heat treatment was below 2%. Its microstructure was isotropic and consisted of martensite and fine homogeneous globulitic carbides, shown in Fig.1. The figures presented show the whole process of preparation, production of HIP and heat treated product, complied with the requirements on pore-free material with a homogeneous microstructure.

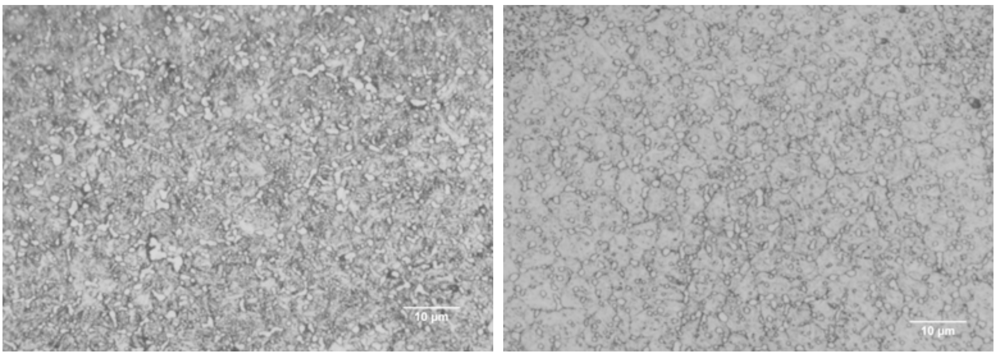

Fig.1. Microstructure of the material M2 + Nb, LM a) HT B b) HT G.

The solidification carbidic phases of the M_6C type were obtained by selective etching by 4% NaOH in a saturated solution of $KMnO_4$ and were used for image processing and statistical analysis.

In publication [16] to characterize the size of carbides we selected an area which represented the size of the most frequently occurring particle from the investigated set. The area proportion of the phases was 14.14% with HTG and 13.48% with HTB. Attending experimental data show up to a 50% decrease in the medium size of an area of analyzed carbide phases with HTG and the same tendency was observed for the medium size of perimeter but with only a 20% decrease. One can assume partial diffusion of Nb into the matrix during austenitization, hardening and tempering which resulted in the refining of microstructure and formation of fine precipitates of construction steel (and thus also increased toughness) [13,16].

Image Processing and Analysis

Digital LOM images of the carbides were pre-processed to remove noise and eliminate a light deviation. The key step of image analysis in general is thresholding. Thresholding is segmenting the image into features of interest and background. In this case it was necessary to remove pores and matrix to get the mask of carbides as shown in Figs.2 and 3. Statistical evaluation of the microstructural parameter requires the measure of a representative amount of objects. Combination of magnification 1000x and high resolution camera mode allowed us to analyze a plane of size approximately 920x120 μm with 100 000-140 000 carbides. Size and shape characteristic of the carbides was measured from a mask of carbides. Planar distribution of carbides in the plane of the cut was evaluated using Voronoi tessellation [17]. Carbides in the mask of pores were replaced by their geometrical centroids. Centroids were used for generation of the Voronoi tessellation. The cells of Voronoi tessellations were measured by image analysis. Statistica 7.2 was used for statistical processing of measured geometrical characteristics.

Size of the carbidic phases was evaluated using area, perimeter and Feret diameter. Values of these parameters were measured directly from thresholded images. Thresholded carbides were fitted by ellipse, the major and minor axis of the ellipse was used as another size characteristic. Size distribution of the carbidic phases after heat treatment G (HTG) is shifted to a smaller value compared to HTB. Shape of the carbidic phases was evaluated by circularity. For exact evaluation of the shape it is necessary to make images at higher magnification. This paper is oriented mostly on planar distribution and the size of carbidic phases. Results of size and shape measurements are shown in Tab.1. The most significant difference between HTG and HTB samples was in the area value.

Planar distribution of carbidic phases was evaluated by measurement of Voronoi tessellation characteristics. The same geometrical characteristics as in the case of size distribution was used, but interpretation of these characteristics is quite different. Area, perimeter and Feret diameter are parameters of Voronoi cells. The Voronoi cell represents free space around the carbide. It necessary to follow up the size of carbides at the same time, because Voronoi tessellation gives no information about size of the carbides. Large carbidic phase and small carbide alone in the matrix give similar results of cell area. The coefficient of variation is an important parameter of Voronoi tessellation. In the case where the cell area of Voronoi tessellation indicates the tendency to regularity, $CV > 0.529$ means tendency to irregularity and cluster making, $CV < 0.529$ means tendency to regularity. From Table 2 there is resulting a tendency to homogeneous distribution of carbides in sample HTG, $CV_{area} = 0.478$. The sample HTB has more irregular carbide distribution CV = 0.522. Heterogeneous carbides distribution of HTB sample in contrast with HTG show maxima values (Max) of all (unless circularity) characteristics as well.

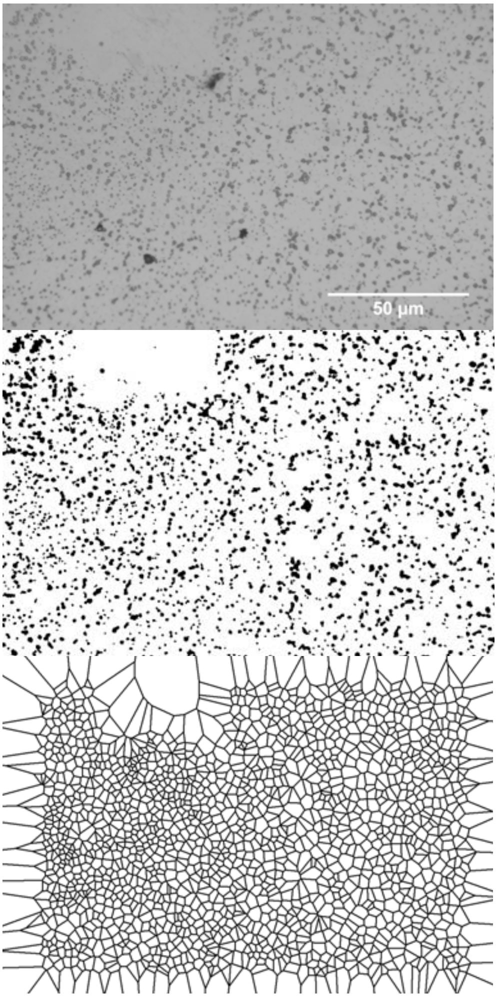

Fig.2. Carbide distribution of HTB sample a) LOM image, b) mask of carbides, c) Voronoi tessellation.

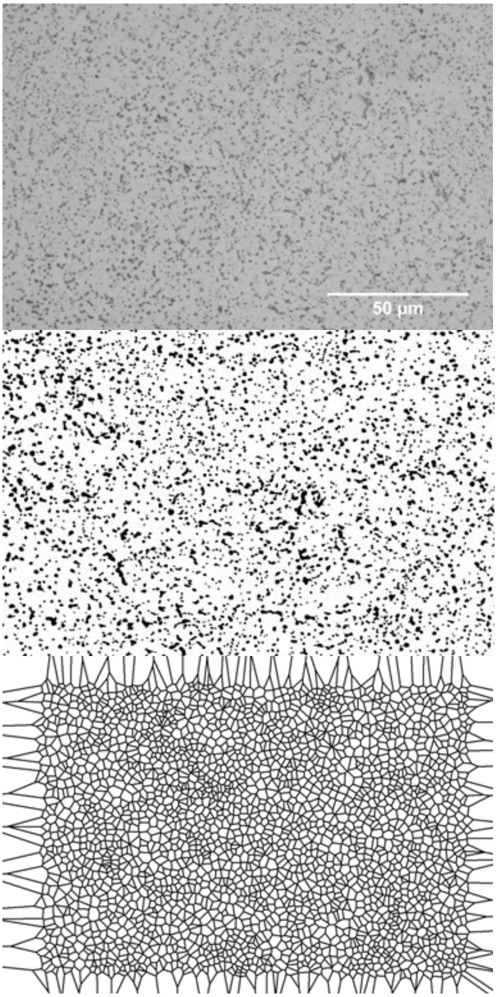

Fig.3. Carbide distribution of HTG sample a) LOM image, b) mask of carbides, c) Voronoi tessellation.

Tab.1. Statistical evaluation summary of size and shape characteristics of the carbidic phases.

		Area [μm²]	Perim. [μm]	Major [μm]	Minor [μm]	Circ.	Feret [μm]
HTB	Mean	1.37	4.59	1.50	0.99	0.77	1.59
	SD	1.40	2.88	0.85	0.40	0.15	0.91
	Min	0.20	1.59	0.51	0.20	0.07	0.55
	Max	33.26	60.38	11.55	5.49	1.00	13.19
HTG	Mean	0.89	3.74	1.26	0.79	0.77	1.34
	SD	0.81	2.15	0.66	0.28	0.16	0.72
	Min	0.20	1.59	0.51	0.19	0.08	0.55
	Max	17.23	35.46	9.45	3.83	1	12.40
Deviation between samples [%]	Mean	53.93	22.72	19.04	25.31	0	18.65

Area – area of carbidic phases; Perim – perimeter of carbidic phases; Major, Minor - are the primary and secondary axis of the best fitting ellipse; Circ. – circularity = $4\pi(\text{Perim}/(\text{Area})^2)$; Feret - Feret's Diameter, the longest distance between any two points along the selection boundary; Deviation between the samples – mean deviation calculated as (HTB-HTG)/HTG; SD – Standard deviation

Tab.2. Statistical evaluation summary of the planar distribution characteristics calculated from Voronoi tessellation generated by carbidic phases.

		Area [μm²]	Perim. [μm]	Major [μm]	Minor [μm]	Circ.	Feret [μm]
HTB	Mean	0.69	3.39	1.13	0.74	0.71	1.27
	SD	0.36	0.81	0.29	0.22	0.08	0.30
	Min	0.01	0.13	0.05	0.05	0.24	0.06
	Max	22.56	18.49	5.84	4.92	1	6.54
	CV	0.522	0.239	0.257	0.297	0.113	0.236
HTG	Mean	0.46	2.75	0.93	0.60	0.72	1.04
	SD	0.22	0.63	0.23	0.17	0.08	0.24
	Min	0.01	0.13	0.05	0.05	0.24	0.06
	Max	3.63	7.71	2.87	1.88	1	3.13
	CV	0.478	0.229	0.247	0.283	0.111	0.231
Deviation between samples [%]	Mean	50.00	23.27	21.50	23.33	1.38	22.15

Area – area of Voronoi tessellation cells; Perim – perimeter of Voronoi tessellation cells; Major, Minor - are the primary and secondary axes of the best fitting ellipse; Circ. – circularity of Voronoi tessellation cells; Feret - Feret's Diameter, the longest distance between any two points along the selection boundary of Voronoi tessellation cells; CV – coefficient of variation; SD – Standard deviation

Hardness

The carrier of high hardness of high speed steels, also at high temperature machining, are carbides of the type M_6C and M_2C (M = W, Mo), which precipitate at tempering temperatures of 550 up to 580°C during the so-called secondary quenching process. The properties of high speed steels are dependent on type and hardness of carbidic phases which are primarily formed during solidification, and secondary carbides that precipitate from solid solution during tempering [18-20]. In steels with a higher content of vanadium there also exists VC which precipitates at the same temperatures. After an overload of optimal temperature a coagulation of resistant carbides occurrs, and so hardness rapidly decreases with temperature increasing [19-22]. High speed steel STN 41 9830 is considered as middle range efficient. Optimal heat treatment is expressed by an increase of hardness of powder high speed steels. It was confirmed by the microhardness measurement HV1 and hardness HRC, which are calculated as a mean value from 15 measurements: 835 HV1, 65 HRC for the G sample in comparison with 736 HV, 62 HRC for the B sample.

Microhardness HV1 for steels with different heat treatments was higher by 100 units in comparison with microhardness value of sample B. To the microhardness values correspond the HRC values with relative lower differences among the heat treatments.

CONCLUSION

- Size of the carbidic phases decrease after heat treatment G, the mean area of carbides in HTG is ~50% smaller as in the HTB sample, mean value of perimeter, fitted ellipse and Ferret diameter is ~20% smaller as well.
- Planar distribution of carbidic phases is more homogeneous in HTG, the coefficient of correlation of mean area CV_{area}=0.478 as compared with HTG CV_{area}=0.522.
- Comparison of size and planar distribution values shows a stronger tendency to regular and homogeneous distribution of carbidic phases after heat treatment G.
- Microhardness and hardness of materials after heat treatment G are higher 835 HV1 and 65 HRC in comparison with HT B 736 HV1 and 62 HRC.

In conclusion we can state that the optimum heat treatment was HT G, austenitizing 1100°C/20 min in a vacuum and subsequent hardening at pressure 5bar in a nitrogen medium; tempering 540°C/3x1h. Selection of suitable heat treatment of the material M2, fine alloyed with Nb, allowed us to reach our aim in terms of refining, and a more uniform distribution of carbide phases, and thus also increased microhardness and hardness of the respective material.

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