X-RAY DIFFRACTION STUDIES OF CARBIDES IN THE SPRAY FORMED AISI M2 HIGH-SPEED STEEL

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Abstract

The present work concerns the study of the crystallography of the carbides presents in the high-speed steel AISI M2 processed using spray forming. High-speed steels have a high hardness resulting from a microstructure, which consists of a steel matrix (martensite or ferrite), in which carbides of different crystal structure and chemical composition are embedded. Samples have been taken at different positions regarding the billet height. From these samples, the carbides were extracted by chemical dissolution. The extracted carbides were analysed using the synchrotron radiation source. The results show that there is a microstructure variation according to the billet height. These microstructures present different carbide morphology and distribution. The X-ray diffraction patterns show the presence of different carbide crystallographic structures. It was identified the $M_{23}C_6$, V_8C_7 in the upper and the bottom billet position. The intermediate position shows the presence of $M_{23}C_6$, MC and M_2C . The Rietveld method showed that the M_2C is a phase that has a hexagonal structure and the lattice parameters suggests that is formed by light and heavy atoms.

Keywords: carbides, high-speed steel, X-ray, synchrotron radiation, Rietveld method.

INTRODUCTION

The macroscopic mechanical properties of the alloys are imposed by the microstructure. Until now, there was not a great interest in the study of the crystallographic structures of carbides present in high-speed steels. In this context, the cutting tool inserts have been used with a thin coated layer, e.g., titanium nitride and titanium carbonitride. Hence, the knowledge of these high-speed steel substrate structures has acquired a major importance.

The spray forming process is a new technology to produce pre-shapes of high alloyed materials with homogeneously distributed elements and small and evenly dispersed precipitates with small grain size. This microstructure results from the rapid solidification and on the considerable extension of the solid solubility.

In the spray forming, the solidification process occurs in two stages: first when the droplet fly (rapid cooling) inside the spraying chamber and after when the droplet is consolidated. The carbides are formed in both stages. During the flight of the droplets, occurs the formation of the MC carbide, where M denotes metallic atoms. When the partially solidified droplets impact against the top surface of the growing billet they undergo a fragmentation, which makes the residual liquid to fill the voids by surrounding the solidified layer. In this stage the MC carbide grows and coalesces.

The AISI M2 high-speed steel has high alloying elements content. For this steel class the alloying elements are V, Cr, Mo, W and C. After casting or spray forming any further thermomechanical treatment aims the formation of a ferritic and or martensitic matrix, i.e., in crystallographic words the matrix is pure or near pure iron with bcc structure and a complex network of metallic carbides. This carbide network is formed by atom migration through the matrix.

Notwithstanding, the structure determination of these carbides is a very hard work. Since these structures were formed by atom migration it is not possible to reproduce them by a controlled process with a determined chemical composition. The solution of this problem is to obtain the carbide by chemical extraction from the steel.

The aim of this study was to identify the crystallographic structure of extracted carbides from spray formed AISI M2 high-speed steel.

EXPERIMENTAL

The chemical composition (mass %) of AISI M2 used in this study was Cr, 3.29; Mo, 4.09; W, 6.19; V, 1.87; Si, 0.22; Mn, 0.18; Ni, 0.034; N, 0.034; C, 1.03 and Fe balance. A 20 cm diameter and 10 cm height billet was spray formed using N₂ as the atomizing gas. This material was annealed at 880 °C for 1 hour. This condition not affects the already formed carbides. The samples were obtained in the upper, central and bottom position in the longitudinal billet direction.

Carbides were extracted by chemical dissolution of the matrix with Berzelius reagent ⁽¹⁾. The duration of the dissolution was 12 hours and the carbides were filtrated using a PTFE filter. The microstructure and chemical composition of the carbides were analyzed by scanning electron microscope – SEM (Phillips, XL30) fitted with energy dispersion spectrometer - EDS. Phase constitution was examined using X-ray diffraction performed with a synchrotron source, with 5.42 keV energy. The Rietveld refinements utilized computer program GSAS.

RESULTS AND DISCUSSION

In the spray formed plant used in this work, the billet top is in contact with a stream of particles and gas. This material portion undergoes a faster cooling rate than the rest of the billet. During solidification it is formed a microstructure where the carbides are dispersed in the matrix. In the central billet region the slower cooling rate allows the atomic migration promoting the formation of the carbide network. In the bottom of the billet, the ceramic substrate acts as an isolation material and the carbide network is analogous to that of encountered at the centre, but the cells are smaller. The microstructure describe above can be see in Fig. 1.

The phases $(Cr_{15.58}Fe_{7.42})_{23}C_6$ and V_8C_7 were identified in the top, see Fig. 2, and the bottom regions of the billet. In the central region it was identified two phases, an unknown phase with very high X-ray diffraction peaks intensities and the VC phase. The different phases found in the billet can be related to the cooling ratios at the particular billet region.



(C)

Figure 1: SEM micrographs of the spray formed AISI M2 high-speed steel showing the disperse carbide in the top of the billet (a) and the carbides cell in the centre (b) and bottom of billet where de cell are smaller than in the centre.



Figure 2: X-ray pattern of extracted carbides of the top region of the billet.

The VC non-stoichiometric carbides occur in the fcc structure (space group, *F* m -3 m), where the vacancies are randomly distributed among the non-metal sites. The V₈C₇ is a long range ordered phase, which is also cubic (space group, P 43 3 2) ⁽²⁾ and has an approximately eight times larger elementary cells than the VC phase. The difference between their X-ray diffraction patterns is the satellite peaks of very low intensity. In the central region of the billet this peaks were not identified. This ordering increases the microhardness and the electric conductivity, but the substitutional elements in this crystalline structure are not yet studied.

The M₂C carbide is a metastable phase and it can be easily decomposed in thermal treatment at high temperature and short time. In the high-speed steel this decomposition occurs in the austenitisation stage. In this stage the M₆C carbide nucleates at the M₂C / austenite interface and the MC carbide inside the M₆C carbide or at the M₆C/M₆C interface.

The hexagonal or orthorhombic crystalline structure of the M_2C carbides is function of the ordered or random distribution of the carbon in metallic carbides that is determinate by the cooling temperature, the kinetic of transformation and carbon concentration. The orthorhombic phases can be identified through the distortion of the metallic sub lattice, which induces a splitting of some peaks, which may be detectable, by synchrotron measurements.

The space group P b c n (orthorhombic structure, Fe_2N type) was used for Rietveld refinement of unknown phase ⁽³⁾. The agreement between the measured

and calculated intensities is illustrated in Fig. 3. The observed intensity data is plotted in the upper field as points. The calculated pattern is show as a solid line. The solid line curved below x-axe represents the difference between observed and calculated intensity.



Figure 3: Rietveld refined diffraction pattern of the extracted carbides.

The differences observed are attributed at the preferential orientation due to the particles shapes and the presence of the amorphous phase. Fig. 4 shows the extracted carbides. In the details is possible to observe that the particles have a plate shape. This shape collaborates with the preferred orientations when the sample is prepared.



Figure 4: SEM micrographs of the extracted carbide in the centre of the billet.

The calculated lattice parameter for the unknown phases is a = 0.4649636 nm, b = 0.674553 nm and c = 0.5116704 nm. Since, this phase is isomorphs whit Mo₂C the differences between these parameters are assigned by the substitution of the Mo by another alloy atoms ⁽⁴⁾. The orthorhombic structure of this carbide is commonly accepted as a disordered structure in transition to a hexagonal structure. The presence of this structure in this material is coherent with the thermodynamic history.

CONCLUSION

Using the Rietveld refinement, it was confirmed that de unknown phase found in central region of the billet is an orthorhombic structure. For this carbide type the usual structure is hexagonal, this structured can be attributed to the thermal treatment of this particular sample.

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