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## Evolution of carbides in cold-work tool steels

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#### ARTICLE INFO

#### ABSTRACT

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Keywords: Cold-work tool steel Microstructure Carbide Electron microscopy Phase identification This study aimed to present the complete history of carbide evolution in a cold-work tool steel along its full processing route for fabrication and application. A sequence of processes from cast to final hardening heat treatment was conducted on an 8% Cr-steel to reproduce a typical commercial processing route in a small scale. The carbides found at each process step were then identified by electron diffraction with energy dispersive spectroscopy in a scanning or transmission electron microscope. After solidification, MC,  $M_7C_3$  and  $M_2C$  carbides were identified and the last one dissolved during hot compression at 1180 °C. In a subsequent annealing at 870 °C followed by slow cooling,  $M_6C$  and  $M_{23}C_6$  were added, while they were dissolved in the following austenitization at 1030 °C. After the final tempering at 520 °C, fine  $M_{23}C_6$  precipitated again, thus the final microstructure was the tempered martensite with MC,  $M_7C_3$  and  $M_{23}C_6$  carbide. The transient  $M_2C$  and  $M_6C$  originated from the segregation of Mo and finally disappeared due to attenuated segregation and the consequent thermodynamic instability.

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#### 1. Introduction

Working on metals at ambient temperature is one of the most common ways to form metallic parts in manufacturing industries. In this context, the improvement of cold-work tool materials can make a significant contribution to the efficiency and reliability in manufacturing industries. Among various materials for cold-work tooling, AISI-D2 steel has been the conventional choice for the inserts in dies [1–3]. This ledeburitic alloy contains a large fraction of hard carbides in a tempered martensite matrix and presents excellent wear-resistance, whereas it suffers from low toughness due to the coarse and brittle carbides [4.5]. 8%Cr-steels have been introduced as the modified version of D2 [4,6]. While conventional D2 contains about 12 wt.% of Cr and 1.5 wt.% of C [7], 8%Cr-steels contain about 8 wt.% of Cr (as the name indicates) and have a C content from 0.8 to 1.2 wt.% [4-6,8-11]. Although they are still ledeburitic, the reduced C and Cr content leads to reduced fraction and size of brittle primary carbides, thus resulting in much improved toughness [4–6,8]. It was also reported that tougher carbides existed in some branches of 8%Cr-steels resulting in improved toughness [8–11]. 8%Cr-steels have better tempering resistance due to the addition of Mo, W, or V, thus they retain higher hardness after tempering in spite of the lower C and Cr content. This is more remarkable after tempering at high temperatures around 500 °C due to enhanced secondary hardening [5]. Therefore, 8%Cr-steels are generally harder and

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tougher than D2. The current trend of stronger work-materials and severe demand for dimensional precision is encouraging the usage of advanced cold-work tool steels such as 8%Cr-steels [12–14]. Therefore, various 8%Cr-steels are being introduced and are substituting D2 in some applications owing to their superior performance [12–14].

In spite of their potential as a new universal grade of cold-work tool steel, studies on their characteristics, especially on microstructural ones [4–6,15], have not been supplied in sufficient amount and detail. A large fraction of the performance of cold-work tool steels should be determined by the distribution and the properties of carbides in them. These microstructural features are relevant to the various processes in the course of fabrication and application of the tool steels. Therefore, it is of great usefulness to characterize the carbides in the full process history in order to understand the microstructure–property relationship and to optimize the process and the consequent microstructure. Although this type of work can be found on a high-speed tool steel [16], it has been hardly documented on cold-work tool steels which have substantially different compositions.

The processing route for typical commercial cold-work tool steels is presented in Fig. 1. They are usually ingot-cast after various melting and refining processes. To break up the coarse and inhomogeneous solidification microstructure, hot deformation such as forging and rolling is conducted. Annealing heat treatment is followed to obtain a soft ferritic matrix with spheroidized carbides. At this softened state, the alloy is machined to various shapes of tools by tool or die makers. Quenching and tempering heat treatment is conducted to obtain the hard martensitic matrix with carbides for final applications as tools or dies. As stated, the alloy is subjected to several steps of thermomechanical and heat



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Fig. 1. Schematic illustration of a typical commercial processing route for the fabrication of tool steels and tools (EAF: electric arc furnace, LF: ladle furnace, VD: vacuum degassing, VIM: vacuum induction melting, QT: quenching and tempering).

#### Table 1

Chemical composition of the	he 8%Cr steel used in	this study (wt. %).

С	Si	Mn	Cr	Мо	W	V	Al	Nb	Fe
1.02	1.15	0.43	7.93	1.20	0.19	0.12	0.25	0.09	Bal.

treatment processes, during which the microstructures continue to change. Especially the carbides transform their types and distributions. In this study, a small-scale simulation of the processing route in Fig. 1 was conducted and the full history of microstructural evolution, i.e. the carbide evolution is described following the sequence of the processes. Discussions are then presented in relation with the evolution of thermodynamic equilibria in the course of the processing route.

#### 2. Material and methods

#### 2.1. Materials and processes

The chemical composition of the 8%Cr-steel used in this study is presented in Table 1. To simulate the solidification or casting process, small (50 g in weight) coupons of the alloy were cast by a plasma arc melting apparatus, in which the commercial product of the same alloy in annealed state was provided as the raw material. 3–4 specimens of cylindrical shape were extracted near the center region of one coupon. They were then submitted to the subsequent processes, i.e. hot deformation, annealing, quenching and tempering. At each intermediate step, a specimen was kept for microscopic analyses. During the hot deformation, an additional specimen was obtained by quenching prior to straining. This procedure was intended to observe the change of the initial cast microstructure after reheating to a high temperature and the following isothermal holding. Owing to the small dimension of the ingot and the consequent fast solidification, macroscopic inhomogeneities were substantially reduced in comparison to commercial ingots, which minimized the dependence of microstructure evolution on the position of the specimen. Only small volumes near the surface showed substantially different microstructures, while they were excluded in the sample volume for the subsequent processes.

The detailed process conditions in the simulated processing route are schematically shown in Fig. 2. The hot deformation was simulated by a hot compression test, in which the specimens were reheated to 1180 °C, held for 10 min and compressed by 50% thickness reduction. For annealing, the compressed specimens were held at 870 °C for an hour, then slowly cooled in the furnace. For quenching, the annealed specimens were austenitized at 1030 °C for 30 min and cooled in air. Tempering was performed in 2 cycles at 520 °C. The isothermal holding time at each cycle was 2 h and the specimens were cooled in air.

#### 2.2. Microscopic analyses

A scanning electron microscope (SEM, JEOL JSM-7001F) was used to observe the microstructures. The specimens were polished following the conventional metallographic procedure and finally etched in Vilella solution which was a mixture of 0.6 g picric acid and 3 mL hydrochloric acid in 97 mL ethanol. To measure the fraction of carbides, the revealed carbides in the SEM micrographs were replicated on transparent films. The replicated carbide images were then submitted to the image analyzing software, Image] [17] for the measurement of fraction.

In order to identify the carbides types, electron backscatter diffraction (EBSD) and energy dispersive spectroscopy (EDS) were used in synchronization, when the carbide sizes were over 0.5 µm. The NordlysNano camera for recording EBSD pattern (EBSP), X-Max<sup>N</sup> EDS detector, and the AZTEC software of Oxford Instruments were used.



Fig. 2. Schematic illustration of the processing conditions for the fabrication of the 8%Cr-steel specimens.

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