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Original Research Paper

Microstructure and mechanical characterization of rapidly solidified Cr-C tool steel: Annealing effects

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ABSTRACT

The effect of isochronal annealing on D2 tool steel powder, rapidly solidified via both Impulse Atomization and Water Atomization, has been evaluated using high-resolution scanning electron microscopy and Vickers microhardness. The amount of supersaturation of the alloying elements inside the retained austenite phase as a function of eutectic undercooling was calculated. The fraction of austenite transformed to ferrite at different annealing temperatures (from 350 °C to 810 °C) was also determined, using Rietveld analysis. The results show that although the particles with larger eutectic undercooling have larger supersaturation of alloying elements within the retained austenite phase, they have a smaller fraction of austenite to ferrite transformation at the temperature in which transformation starts. The maximum hardness was achieved at an annealing temperature of 550 °C, due to the formation of fine and well-distributed carbides.

1. Introduction

D2 tool steel is a well-known high-chromium and high-carbon ferrous alloy that can be found in many industrial applications. This material has a high volume fraction of carbides, which results in good wear and abrasion properties. The $(Fe,Cr)_7C_3$ carbide has a hardness in the 1300–1800 HV range [1]. For wear applications, a conventional tool steel casting process is used to make D2 tool steel components, due to the resulting and beneficial coarse carbide structure. For other applications, D2 tool steel is normally cast using Rapid Solidification (RS) techniques [2]. This ensures the formation of a refined microstructure [3].

Although D2 tool steel is of high commercial significance, there have been relatively few studies that characterize its microstructure during RS processing. Two studies reported results from material rapidly solidified using splat quenching and melt spinning [4,5]. Two others used X-ray diffraction and transmission electron microscopy [6,7] to show that RS of D2 tool steel results in the formation of a supersaturated metastable retained austenite phase, and that subsequent annealing results in the formation of rod-like carbide precipitates in a ferrite matrix.

During rapid solidification of tool steels, alloying elements do not have time to diffuse out of the austenite structure but remain supersaturated within it. These elements then precipitate out during subsequent annealing in the form of M_7C_3 carbides. The supersaturation of M_7C_3 carbides ($S_{M_7C_3}$) at a given annealing temperature is defined as [8]:

$$S_{M_7C_3} = \ln\left[\frac{X_M^7 \cdot X_C^3}{K_{M_7C_3}}\right] \quad (1)$$

where X_M and X_C are the supersaturated concentrations of M (Cr, V, etc.) and C in mole fraction, and $K_{M_7C_3} = X_{eM}^7 \cdot X_{eC}^3$ is the equilibrium solubility product given by the equilibrium concentrations of M and C (denoted as X_{eM} and X_{eC}). Temperature-dependent equilibrium solubility products can be determined using a Calphad-type thermodynamics software package. The value of $K_{M_7C_3}$ in D2 tool steel, calculated using Thermo-calc [9], is:

$$\log K_{M_7C_3} = 19.1 - \frac{18,639}{T} \quad (2)$$

The research presented in this study forms the second of two recent studies in our research group examining the microstructural development of D2 tool steel produced via two rapid solidification methods: Impulse Atomization (IA) and Water Atomization (WA). The first study [10] evaluated the effects of the atomization process conditions on the rapidly solidified microstructure in the as-solidified state. From this study, it was shown that higher cooling rates result in a lower percentage of eutectic fraction.

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Consequently, the retained austenite would be supersaturated in carbide-forming elements. A summary of the experimental data from [10] linking eutectic fraction, cooling rate, and undercooling is given in Table 1. In the present study, the effect of annealing temperature on the microstructure of D2 tool steel is explored. Together, the two studies provide a comprehensive analysis of microstructural development during rapid solidification processing of D2 tool steel.

2. Experimental

D2. tool steel is a high-carbon, high-chromium ferrous alloy with 1.55% C, 11.8% Cr, 0.40% Mn, 0.80% Mo, and 0.80% V (all in wt.%). The corresponding pseudobinary phase diagram has liquidus and eutectic temperatures of 1394 °C and 1270 °C, respectively [7], and is given in the first study [10]. In order to investigate the effect of annealing on the microstructure of RS D2 tool steel, particles produced using IA [11] and WA [12,13] were given an isochronal anneal for 2 h at temperatures of 350 °C, 450 °C, 550 °C, 650 °C, 750 °C, and 810 °C in a protective nitrogen atmosphere. 810 °C was chosen as the highest annealing temperature to avoid the initiation of the ferrite-to-austenite phase transformation occurring at higher temperatures, as identified by Thermo-Calc. The heating and cooling rate prior and post anneal was ~0.33 °C/min. Although the cooling rate is relatively low for studying phase transformations, the isochronal anneal was carried out in a differential scanning calorimeter (owing to the size of the powder particles), and the captured cooling curves did not show any peaks related to other reactions during the cooling cycle. Thus, it can be inferred that additional reactions did not take place during cooling.

The powder from IA was generated using an apparatus available at the University of Alberta [11]. First, a bulk alloy was melted and held in an alumina crucible for about 30 min at 1600 °C. Then, the liquid was pushed through orifices at the bottom of the crucible to create droplets via atomization. The falling liquid droplets were then cooled in both helium and nitrogen atmospheres having a maximum oxygen content of 8 ppm, and thus solidified before reaching the bottom of the atomization chamber. The production parameters and resulting powder sizes are shown in Table 2. The powder from WA was provided by the Hoeganaes Corporation (Cinnaminson, New Jersey, USA). The atomized particles were then annealed in a Setaram Labsys Evo Differential Scanning Calorimeter.

IA particles in the size ranges of 300–355 μm and 600–710 μm, solidified in both He and N_2, and WA particles in the size ranges of 90–110 μm and 300–355 μm were subsequently prepared for scanning electron microscopy (SEM), X-ray diffraction (XRD) and neutron diffraction (ND). IA in He provides significantly higher cooling rates during RS as compared to N_2 due to the higher thermal conductivity. Samples of the chosen particle sizes were mounted in epoxy, polished, and carbon coated. First, SEM images of the microstructure were acquired using a JEOL 6301F, a Hitachi S-2700, and a Zeiss EVO LS15. Different instruments were used due to machine scheduling and availability. The diameter of the precipitated carbides was measured from the SEM images using the ImageJ software [14], while compositional variations were measured via EDX analysis on the Zeiss SEM that was equipped with a Bruker silicon drift detector. As carbide particles are irregular in shape, the term diameter refers to an average diameter based on several random measurements made of diagonal distances along the particle's cross-section. Second, XRD was performed using Co-Kα radiation in a Rigaku Denki Rotaflex RU-200B X-ray system for phase identification. High Energy XRD (HEXRD) was also performed, at Argonne National Laboratory. Third, ND was carried out using a neutron beam of 1.33 A° wavelength at Atomic Energy of Canada Limited (AECL) in Chalk River, ON. Lattice parameters were extracted using GSAS software. Finally, Vickers microhardness measurements were performed on the as-atomized and annealed particles using a Buehler Tukon 1102 Vickers hardness tester. Thirty data points were collected for each measurement using a load of 100 gf and a dwell time of 10 s.

Table 1
Experimental data from [10] linking eutectic fraction, cooling rate, and eutectic undercooling during RS.

Atomization technique	Atomization atmosphere	Cooling rate (°C/s)	Eutectic undercooling (°C)	Eutectic fraction (–)
IA	He	7622	68	11
		1589	64	11.4
		538	54	14
IA	N_2	1778	62	12.5
		434	58	13.5
		212	30	19
WA	N/A	111,945	84	8.3
		16,841	72	10.5
		1416	68	11.4

Table 2
IA run conditions and atomized particle size distribution.

Atomization technique	Atomization atmosphere	Number of orifices	Orifice size (μm)	D50 (μm)	σ
IA	He	37	420	670	1.16
	N_2	37	400	510	1.22

$\sigma = \frac{D84}{D50}$.

3. Results and discussion

3.1. Qualitative description of the microstructure

The XRD results, performed on particles between 600 and 710 μm in size produced via IA in N_2, are given in Fig. 1. The spectra for both the initial as-atomized particles and the particles that were subsequently annealed for 2 h at temperatures between 350 °C and 810 °C are shown. As can be seen in the as-atomized spectrum, the as-atomized powders consist of the austenite phase. Thus, the rapid solidification of D2 tool steel resulted in supersaturated metastable retained austenite and the ferrite phase, in

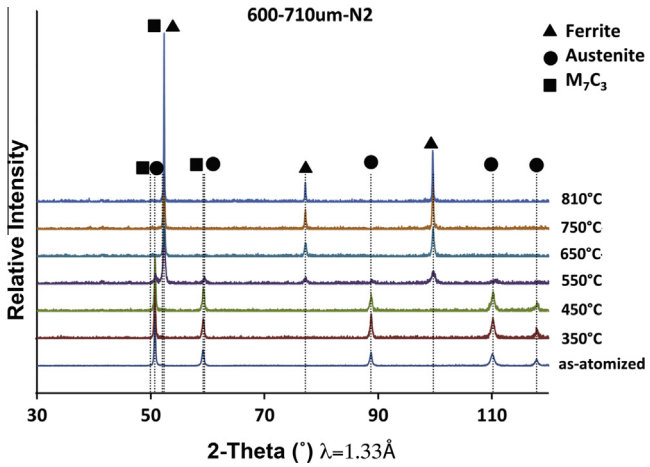

Fig. 1. XRD patterns for IA 600–710 μm powder particles atomized in nitrogen and then annealed for 2 h at different temperatures between 350 °C and 810 °C. Note that for the as-atomized particle, the cooling rate during RS was 434 °C/s.