Effect of solidification rate on microstructure and primary carbides of AISI DC 53 cold work tool steel

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Abstract: The solidification behavior of AISI DC 53 cold work tool steel was investigated by means of a cooling curve and its first derivative. Copper and sand wedge-shaped molds were used to obtain various solidification rates. To reveal the cooling rate degree during solidification, the secondary dendrite arm spacing of the steel alloy was examined by scanning electron microscopy (SEM). The solidification rates of each section for both wedge steel samples were calculated by means of the secondary dendrite arm spacing using a research-based empirical relation from the literature. Experiment results revealed that at the tip region of the cast specimen in the copper wedge-shaped mold, the carbide size was 7–8 μm , where the solidification rate was approximately $4,830\text{ }^{\circ}\text{C}\cdot\text{s}^{-1}$. The greatest carbide size obtained in the upper region of the sand cast wedge-shaped specimen was 250–270 μm .

Key words: copper and sand wedge-shaped mold; AISI DC 53; high solidification rate

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High-chromium cold work tool steels are employed for cold work applications involving surface temperatures of not more than $200\text{ }^{\circ}\text{C}$, such as coining and punching^[1]. In this temperature range, the steels must have high hardness, high wear resistance and toughness, and high dimensional stability during hardening and tempering. Conventional production methods involving thick section casting of cold work tool steels result in low solidification rates. Low solidification rates cause segregation of alloying elements, and large and coarse interdendritic primary carbide structures after solidification. The main drawback of these steels is the nonhomogeneous microstructure because of the high segregation of alloying elements during solidification^[2].

Once solidified, a segregated microstructure is very resistant to subsequent heat treatment (quenching and tempering), and steels with segregated microstructures have brittle fracture and anisotropic properties that are problematic^[3]. Primary carbides are difficult to dissolve into the tool steel matrix by heat treatment. Conventional tool steel production techniques, such as ingot casting and subsequent alternating solutionizing and forging

cycles, have been applied to dissolve and refine the primary carbides. However, it is more convenient to prevent their formation by processes having fast solidification rates.

Individual coarse carbides with irregular and rounded shapes increase the stress concentration at the matrix/carbide interface. The carbide clusters cause microcracks in cold work tool steels, and the coalescence and growth of these microcracks cause brittle failure during loading of these materials. To avoid this type of brittle fracture of the tool steels, the primary M_7C_3 ($\text{M} = \text{Cr}, \text{Mo}, \text{V}$) carbides must be refined, spheroidized and homogeneously distributed^[4]. Therefore, the toughness of the steel can be improved. To achieve this increased toughness, elemental segregation must be lowered by increasing the solidification and cooling rate.

There are some studies regarding the fast and rapid solidification of high-chromium cold work tool steels^[5-7]. Fast solidification provides an ultrafine microstructure by substantially lowering the segregation during solidification^[8-9]. Such a phenomenon brings attention to the importance of the solidification rate on the microstructure during the casting of these steels.

The aim of the present work is to evaluate the solidification behavior of a new type of cold work steel, AISI DC 53^[10], by means of computer-assisted cooling curve analysis. Then, the effect of cooling rates on the microstructure, especially the primary carbide size of

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these steels, can be analyzed. For this purpose, a series of tool steel alloys were melted and poured into different molds: sand and copper wedge-shaped molds.

1 Experimental procedure

The chemical composition of the tool steel used in this study is listed in Table 1. The melt of the tool steel was prepared using an induction furnace under air atmosphere. Then, the melt was poured at 1,550 °C into a copper wedge-shaped mold. To completely fill the mold cavity and to prevent early freezing during pouring, the copper wedge-shaped mold was preheated to 200 °C. For comparison, the melt was poured into a wedge-shaped sand mold cavity under the same conditions. The wedge samples were cut to obtain three cross-section samples for the upper, middle and tip regions, as shown in Fig. 1. Picral solution was used as an etchant for all the wedge samples. Microstructural examinations were performed by scanning electron microscopy (SEM) to examine the secondary dendrite arm spacing and carbides. Carbide extraction was performed by leaching the tool steel in HCl acid with 37% purity to measure the carbide size. The carbide size was also measured by SEM analysis techniques. The quantitative analysis of the retained austenite was performed by X-ray diffraction (XRD) using Cu K α radiation at 40 kV and 30 mA with a step size of 0.02° at a scanning rate of 2 °·min⁻¹.

Table 1: Area fraction of primary α -Al in Al-10Si modified with different amounts of Al-5Ti (wt.%)

C	Cr	Mn	Si	Mo	V	Fe
0.9	7.8	0.5	0.9	2.5	0.5	Bal.

Fig. 1: Schematic diagram of casting specimen

During castings, thermal analysis was carried out to reveal the liquidus temperature and the carbide start and finish temperatures by pouring liquid steel into the resin-bonded Quik-cups with a Pt-10%Rh thermocouple placed in the center of the cup. The temperatures were recorded against time using an Elimko E630 data logger for further cooling curve analysis. The data recorded were processed by MATLAB 2008 to obtain the first derivatives

to determine the phase transformation temperatures of AISI DC 53 cold work tool steel. Microstructural examinations were carried out by optical microscopy and XRD on the sample taken from the thermal analysis cup close to the hot junction of the thermocouple. The thermal analysis sample was etched by Vilella's reagent, and XRD analysis of the sample was performed using Cu K α radiation at 40 kV and 30 mA with a step size of 0.02° at a scanning rate of 0.02°·min⁻¹. Carbide extraction was performed on the same sample by leaching tool steel in HCl to reveal the carbide type.

2 Results and discussion

2.1 Solidification analysis

The cooling curve (Fig. 2) shows that the solidification of AISI DC 53 cold work tool steel starts by the nucleation and growth of the austenite phase at approximately 1,418 °C. Solidification terminates with a 100% austenite phase. At the final stage of the solidification, due to segregation, carbon and chromium saturate at grain boundaries and initiate interdendritic primary carbides with a skeletal morphology. The start and finish temperatures of forming these carbides were obtained from the first derivative of the cooling curve. The first peak on the first derivative curve corresponds to the liquidus temperature (1,418 °C) on the cooling curve. The second peak on the first derivative curve corresponds to the carbide start temperature (1,148 °C) on the cooling curve, and the last inflection point on the first derivative curve represents the carbide finish temperature (850 °C) on the cooling curve.

Fig. 2: Cooling curve and first derivative of AISI DC 53 tool steel

The diffraction pattern of the thermal analysis sample taken from the middle section close to the thermocouple hot junction point is given in Fig. 3. The retained austenite and bct martensite peaks can be seen in the diffraction pattern. Figure 4 shows the microstructure of the tool steel solidified in the thermal analysis cup. The light phase is primary retained austenite, and the dark regions are bct martensite. The interdendritic phase, which exhibits a skeletal morphology at the austenite grain boundaries saturated by carbon and chromium, is likely to be the carbide phases. XRD analysis of these carbides extracted by leaching tool steel shows that the carbides are Cr-based M₇C₃ carbides (Fig. 3).

Fig. 3: XRD pattern of AISI DC 53 steel specimen cast into thermal analysis cup

Fig. 4: As-cast microstructure of AISI DC 53 alloy poured into a thermal analysis cup showing dark martensite and light austenite phases surrounded by carbide phases

2.2 Microstructural analysis of wedge-shaped castings to reveal cooling rates

SEM microstructures of tool steel solidified into the sand and copper wedge molds are given in Figs. 5 and 6, respectively. The dendritic light gray phase is bct martensite, and the dark regions are primary retained austenite. The secondary dendrite arm spacing of tool steel was measured using the SEM images. The greatest secondary dendrite arm spacing was found in the region of the middle section (close to the upper section) of the sand cast wedge sample (32.94 μm), and the smallest secondary dendrite arm spacing was found in the tip section of the copper cast wedge sample (7.64 μm). In the upper section of the sand cast wedge sample, the equiaxed dendritic structure can be seen in the microstructure. The secondary dendrite arm spacing (SDAS) of the sand and copper wedge samples showed an approximately linear relationship with respect to the section thickness, as shown in Fig. 7. The slope of the SDAS versus section thickness curve for the copper wedge sample (1.44×10^{-4}) steel is lower than the slope of the wedge sample cast into the sand mold (2.19×10^{-4}).

The SDAS versus section thickness relation was determined as follows:

$$(a) \text{ Sand casting SDAS} = 8.27 + 2.19 \text{ ST}$$

$$(b) \text{ Copper permanent mold casting SDAS} = 1.72 + 1.44 \text{ ST}$$

where ST = section thickness.

The main reason for the difference between the slopes is the low thermal conductivity of sand with respect to that of copper. The SDAS of the wedge shape tool steel specimen cast into sand mold was found to be greater than the SDAS of the wedge

Fig. 5: Dendritic microstructures taken from three different sections of sand cast wedge sample: (a) upper (thick), (b) middle, and (c) tip section

Fig.6: Dendritic microstructures taken from three different sections of permanent copper mold cast wedge sample: (a) upper (thick), (b) middle, and (c) tip section

Fig. 7: SDAS versus section thicknesses measured on sand and copper permanent mold cast specimens

Fig. 8: Variation of cooling rate versus section thickness of as-cast tool steel solidified in sand and copper molds

shape specimen cast into copper mold with the same section thickness due to the lower thermal conductivity of silica sand compared to that of pure copper.

In addition, the cooling rates of the different sections of wedge specimens solidified in the sand and copper molds were calculated using the empirical relationship of SDAS given in Ref. [11] at various cooling rates (C_R) and steel carbon contents ($W[C]$):

$$SDAS = 143.9 \cdot C_R^{-0.3616} \cdot W[C]^{(0.5501 - 1.996W[C])} W[C] > 0.15$$

The cooling rates of the sand and copper wedge samples showed an approximately second-order exponential relationship with respect to the section thickness, as shown in Fig. 8.

The cooling rate vs. section thickness relationship was determined as follows:

(a) Sand casting:

$$C_R = 1107.96 - 161.10 ST + 6.06 ST^2$$

(b) Copper permanent mold casting:

$$C_R = 7940.12 - 1092.59 ST + 37.39 ST^2$$

where C_R is the cooling rate.

The lowest cooling rate was found in the region of the middle section (close to upper section) of the sand cast wedge sample (approximately $85^\circ\text{C}\cdot\text{s}^{-1}$), and the highest cooling rate was found in the tip region of the copper cast wedge sample (approximately $4,380^\circ\text{C}\cdot\text{s}^{-1}$).

2.3 Retained austenite determination

The volume fraction of the retained austenite of steel was determined by using a Rigaku X-ray diffractometer using $\text{Cu K}\alpha$ radiation based on the integrated intensity of the martensite and austenite peaks (direct comparison method) using the following equation^[12]:

$$I_{\text{austenite}}/I_{\text{martensite}} = \frac{R_{\text{austenite}} \cdot C_{\text{austenite}}}{R_{\text{martensite}} \cdot C_{\text{martensite}}}$$

where $I_{\text{austenite}}$ is the integrated intensity obtained at the (220)

austenite peak and $I_{\text{martensite}}$ is the integrated intensity obtained at the (200) martensite peak of the copper and sand wedge steel samples. $R_{\text{austenite}}$:65.92 and $R_{\text{martensite}}$:33.55 are the multiplicity factors calculated for the (220) austenite peak and (200) martensite peak, respectively. $C_{\text{martensite}}$ and $C_{\text{austenite}}$ are the volume fractions of the retained austenite and martensite, respectively.

The volume fractions of retained austenite in the tip and middle regions of the copper wedge steel sample are approximately 64.26% and 39.98%, respectively, and the volume fraction decreases to 30.72% in the upper region of the copper wedge steel sample. The volume fraction of the retained austenite in the tip and middle regions of the sand wedge steel sample is approximately 45.43% and 27.66%, respectively, and decreases to 20.88% in the upper region of the sand wedge steel sample.

High solidification rates caused the supersaturation of austenite with carbon and alloying elements, resulting in a decrease in the martensite start temperature [8-10]. After solidification, a large amount of metastable (or residual) austenite (in the range 64.26%–30.72% for the copper mold and 45.43%–20.88% for the sand mold) was present in the tool steel matrix of both samples at room temperature. In addition, it can be observed that the retained austenite content increases with increasing solidification rate, as shown in Table 2.

Table 2: Retained austenite determination and cooling rates for different wedge sections

Wedge sections	Retained austenite volume percentage	
	Copper mold	Sand mold
Tip region	64.26	45.43
Middle region	39.98	27.66
Upper region	30.72	20.88

2.4 Carbide size distribution

The carbide size was determined by SEM images of the extracted carbides by 37% HCl acid of each region of the copper and sand wedge steel samples. The carbide size of AISI DC 53 tool steel at the tip region and middle region for the copper wedge steel sample is approximately 7.13 μm and 44.02 μm , respectively, and increases to 97.72 μm in the upper region. The carbide size of AISI DC 53 tool steel at the tip and middle regions for the sand wedge steel sample is approximately 32.16 μm and 102.43 μm , respectively, and increases to 262.70 μm in the upper region. Figure 9 shows the SEM image of the extracted carbide of the tip section taken from the sample cast in copper mold after HCl acid leaching treatment. The average carbide size of the tip region is 7–8 μm .

In the case of high solidification rates, the nuclei of the carbides increase, and the primary carbides are refined. It can be seen that the carbide size decreases with increasing solidification rate of a particular section in the wedge shape cast tool steel, as shown in Table 3.

Fig. 9: Carbide extracted from tip section of tool steel cast in copper mold

This study states the extreme solidification rates of AISI DC 53 cold work tool steel from a high solidification rate of a copper wedge-shaped mold to a slow solidification rate of a sand wedge-shaped mold. In industry, this type of tool steel is cast into ingot iron molds. In other words, the solidification rate of this commercial steel is moderate between the copper and sand wedge-shaped mold solidification conditions. Therefore, as-cast industrial AISI DC 53 tool steel has a microstructure between fully dendritic (tip region of the copper wedge-shaped mold) and equiaxed (thickest region of the sand wedge-shaped mold). Additionally, the carbide size and retained austenite amount of as-cast commercial AISI DC 53 are between the two extreme solidification rates stated above. For this reason, this study can be a guide for engineers and workers for subsequent processes, such as rolling, annealing and heat treatment of AISI DC 53 tool steel.

Table 3: Carbide size distribution against section thickness in wedge specimens produced by both sand and permanent mold casting after carbide extraction

Copper wedge-shaped mold	
Section thickness	Average carbide size
4.42 – 8.27 mm (tip)	7.13 μm
8.79 – 13.02 mm (middle)	44.02 μm
13.54 – 18.24 mm (upper)	97.72 μm
Sand wedge-shaped mold	
Section thickness	Average carbide size
2.37 – 6.58 mm (tip)	32.16 μm
6.75 – 11.56 mm (middle)	102.43 μm
11.73 – 16.07 mm (upper)	262.70 μm

3 Conclusions

Copper and sand wedge-shaped molds were employed to study the solidification behavior of AISI DC 53 cold work tool steel, which provided different solidification rates. The secondary dendrite arm spacing and size distribution of the carbides formed under these solidification rates were studied with SEM. The retained austenite volume percentage of the samples was measured. The following conclusions have been reached:

(1) The secondary dendrite arm spacing of sand and copper wedge steel samples showed an approximately linear relationship with the section thickness.

(2) The cooling rates of the sand and copper wedge samples showed an approximately second-order exponential relationship with the section thickness.

(3) High solidification rates cause a large amount of metastable (or residual) austenite present in the steel at room temperature, and the amount of retained austenite decreases with increasing solidification rate.

(4) High solidification rates cause the primary carbides to nucleate. Moreover, the existent solid phases do not have sufficient time to grow and they became fine and homogeneously distributed. The primary carbide size decreases with an increasing solidification rate.

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