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Effect of directional solidification of electroslag remelting on the microstructure and primary carbides in an austenitic hot-work die steel

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ABSTRACT

The microstructure of as-cast ingot and three-dimensional microstructure of carbides were analyzed by optical microscope and scanning electron microscope. The types of carbides were identified by X-ray diffraction. Directional solidification of electroslag remelting effectively reduced the segregation of alloying elements in as-cast ingot. The growing direction of dendrites in as-cast ingot refined by directional solidification of electroslag remelting was paralleled to < 001 > crystallographic orientation. The solidification microstructure of austenitic hot-work die steel was composed of austenite and primary carbides (V_8C_7-type and Mo_2C-type) which distributed along grain boundaries. Compared with conventional electroslag remelting, the directional solidification of electroslag remelting process reduced the size of primary carbides and improved dispersed distribution of carbides, but not changed the types and compositions of carbides. The direction of driving force for carbides growth was irregular in conventional electroslag remelting, while that was nearly parallel to crystal < 001 > in directional solidification of electroslag remelting.

1. Introduction

The working temperatures for molding cavities served as copper extrusion die range from 700 °C to 900 °C. The service conditions of extruding dies for copper alloys are very rigorous. Conventional hot-work die steels are widely applied in hot forging, hot extrusion and die casting. In fact, hot-work die steels with martensite matrix, including 3Cr2W8V, H13, THG2000 and QRO90, are widely used in industrial applications (Li et al., 2015). Zhou et al. (2011) reported that the temperature at the surface of dies may reaches up to 600 °C or exceeds the tempering temperature of steel, inevitably leading to the cumulative effect of tempering and certainly affect properties of dies such as hot hardness, temper resistance and high-temperature fatigue strength.

In order to prevent the decrease of strength and the premature failure caused by coarsening carbides and the recovery of martensite, a high-strength insulating coating on the functional surface of die steels (such as a layer of zirconium oxide) was employed, but it has been confirmed to be invalid for service temperature above 600 °C (Grabovskii, 2000). In recent years, austenitic hot-work die steels have attracted researchers' attentions in order to improve the strength of die steels at the temperatures higher than 600 °C. Xie et al. (1990) reported that the more austenite there was in the matrix, the better was the hot strength for hot-work die steel at elevated temperature. Baglyuk et al. (2006) suggested that the possibility of using powder metallurgy to prepare effective die steels that exhibit increased hardness and heat resistance at temperature not less than 850 °C. Wang et al. (2015) investigated effects of B on high temperature mechanical properties and thermal fatigue behavior of austenitic die-casting die steel, they found that thermal fatigue resistance of austenitic die steel was much better than that of martensitic die steel H13.

The microstructure of austenitic hot-work die steels keeps a stable austenite phase, and there is no phase transformation during high temperature services. Therefore, these die steels have longer service life and better thermal stability, but lower hardness. These steels are strengthened by solid solution strengthening and precipitation strengthening with intermetallic, carbides and carbonitrides phases. The stable austenitic structure is composited by Fe-Cr-Ni, Fe-Cr-Mn and Fe-Mn-C alloy system. Grabovskii and Kanyuka (2001) reported that austenitic hot-work die steels ÉK39 and ÉK40 provided by Fe-Cr-Ni matrix have good hot-hardness, good impact toughness and good thermal stability, and these steels can preferably meet requirements of extrusion for copper at service temperature above 700 °C. The elements such as carbon, nitrogen and nickel, are austenite phase forming elements. At the same time, nitrogen is always added to form VN, CV and Cr_2N precipitations to provide precipitation strengthening (Wang et al., 2010.). In order to save precious nickel, manganese is used to replace nickel to obtain stable austenitic matrix. Austentic hot-work steel with low thermal conductivity, tends to produce solidification mushy zone in

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the casting process, which will lead to the center segregation, porosity and the shrinkage cavity.

Electroslag remelting (ESR) could significantly improve the cleanliness, solidification structure, and transverse mechanical properties of steel (Shi et al., 2013). However, the increasing demands on steel mechanical properties urge metallurgists to make greater efforts to eliminate the defects of steel microstructure, such as shrinkage and segregation. Fu et al. (2015) demonstrates that the combination of directionally solidification technology with electroslag remelting technology effectively eliminates macro-segregation in as-cast ingot through the shallow molten metal pool controlled by directional solidification. Compared to ESR process, the amount and size of inclusions in ESR-CDS process could be reduced. Shallow flat pool caused by ESR-CDS process was more conductive to the removal of inclusions. Meanwhile, smaller secondary dendritic spacing and smaller non-equilibrium precipitated phases could be controlled by a higher cooling rate in ESR-CDS process. High cooling rate with spraying water at bottom of ingots could provide stable temperature gradient nearly parallel to axis of ingots (Li et al., 2016). In recent years, the studies on the directional solidification of ESR were focused on the production of superalloy. In this work, the effect of directional solidification of ESR on the segregation, microstructure and primary carbides evolution in as-cast ingot was studied. Moreover, the mechanism of the effect of directional solidification of ESR on primary carbides distribution and morphology was discussed.

2. Experimental

2.1. Experimental materials

The austenitic hot-work die steel was obtained by melting pure alloy ingredients in a 200 kg vacuum induction furnace. The liquid steel was cast into a rod, and then forged into two rods of 120 mm in diameter. Then the electrodes were remelted using conventional ESR and ESR-CDS for comparison. The remelting process was conducted in the argon gas atmosphere. The produced as-cast ingots of 160 mm in diameter remelted by ESR and ESR-CDS were sampled as S1 and S2, respectively. The chemical compositions of remelted ingots S1 and S2 were determined by inductively coupled plasma optical emission spectrometer, and the results are shown in Table 1.

2.2. Microscopic observation

The samples with the dimension of 15 mm × 15 mm × 12 mm were taken from as-cast ingots S1 and S2, respectively. The metallographic samples were analyzed by optimal microscope (LEICA DM2500M, OM) and scanning electron microscope (FEI MLA250, FEI, Hillsboro, OR, USA, SEM), after grinding, polishing and etching with 6% nitric acid alcohol. The precipitated phases in austenitic hot-work die steel were calculated by Thermo-Calc software.

2.3. Carbides collection using electrolytic extraction technique

The samples taken from S1 and S2 ingots, were machined into a rod of Ø15 mm × 90 mm. Carbides were extracted from steel matrix in organic solution (methanol, tetramethylammonium chloride, glycerin, diethanol amine) by electrolysis. Some of the carbides were analyzed by XRD (Rigaku D_{max}-RB, Rigaku, Tokyo, Japan) to confirm the types, and some carbides were observed by SEM for the three-dimensional morphology.

Table 1
Chemical composition of steel (wt/%).

	C	Si	Mn	Cr	Mo	V	P	S	Fe
S1	0.70	0.55	14.95	3.45	1.57	1.723	0.0085	0.0023	Bal.
S2	0.698	0.544	14.90	3.53	1.55	1.726	0.0088	0.0021	Bal.

3. Results and discussion

3.1. Macrosegregation of as-cast ingot

Eliminating macro-segregation in as-cast ingots was a critical factor for precipitation-hardened steel used at elevated temperature (Zhou et al., 2013). Inhomogeneous structure owing to the segregation of alloying elements during solidification played an important role on impact toughness of cast steel (Lan et al., 2000). Auburtin et al. (2000) showed that the flow of solute-rich interdendritic liquid in the mushy zone during solidification was responsible for most types of macro-segregation such as freckles. The freckles were common in ESR ingots on account of deep molten pool with steeper sides, large mushy zones and long local solidification time. During the conventional ESR process, the cooling rate was slow relatively, opposing thermal and solute buoyancy forces led to the remelting and the plume in the mushy zone, and then the segregation channel formed. The deep V-shaped pool profile with a prolonged mushy zone enhanced the intensity of liquid flow in an ESR ingot, so the macro-segregation was more likely to be formed. The ESR-CDS process was developed on the base of the traditional ESR technique. In ESR-CDS process, the macro-segregation was effectively eliminated because of the shallow metal pool with a uniformly distributed mushy zone. At the same time, the severe segregated region existing in the interfaces of columnar grains with different orientations in center of ESR ingot was eliminated. Fig. 1 presents the principle diagram of ESR-CDS, and the longitudinal macrostructure of the ESR-CDS ingot is shown in Fig. 2.

Fig. 2 presents the macrostructure of S2 ingot showing columnar crystals growing nearly parallel to the axis. The direction of some columnar grains deviated a little from the axis of the ingot, and the maximum deviation was about 13°. Deviation of the growth direction may be due to the temperature fluctuation at the solid/liquid interface. It could be seen that the ESR-CDS technique completely eliminated the solidification mushy zone in as-cast ingot. The element segregation was reduced, and the homogeneity of the structure was improved. In addition, the growing direction of columnar grain was nearly parallel to < 001 > crystallographic orientation. Li et al. (2016) pointed out that this parallel oriented columnar grain boundaries avoided the maximum shear stress direction of 45° to the axis of ingot during deformation, which greatly improved hot-workability. While in conventional ESR ingot, there were coarse equiaxed grains and interfaces of columnar grains with various orientations, so processability of ingot with non-oriented columnar grain obviously worsened. The direction of maximum shear stress was consistent with the slip crystallographic orientation of {111} < 110 > slip system during longitudinal

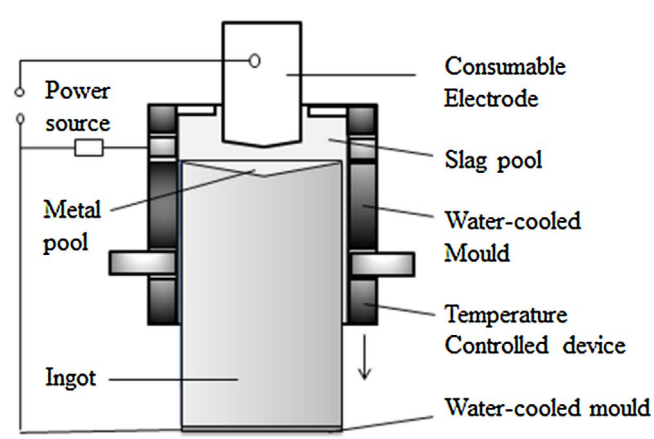

Fig. 1. Schematic diagram of ESR-CDS.