Morphological Evolution during Partial Re-Melting of AISI D2 Cold-Work Tool Steel (Perubahan Morfologi Keluli Kerja Sejuk AISI D2 melalui Peleburan-Semula-Separa)

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ABSTRACT

The impact of isothermal temperature and holding time on the microstructural evolution process is studied. This process is used to produce globular microstructures during partial re-melting of AISI D2 cold work tool steel. The experimental results showed that the optimum process factors should be selected according to the criteria for average grain size and shape factor and also by acknowledging the distribution of the liquid phase around the grains. With increasing isothermal temperature and holding time, the liquid phase starts to diffuse into the grain boundaries and spread more evenly. Also, the equiaxed grains were transformed into near-globular shape, which makes the grain size and shape factor more favourable for thixoforming. The work shows that the optimal process condition for the thixoforming of AISI D2 is 1340°C and 5-min holding time, having a shape factor of 1.13 and a particle size of 51 µm.

Keywords: AISI D2; direct partial re-melting; microstructure evolution; thixoforming

ABSTRAK

Kesan suhu dan masa memegang proses evolusi mikrostruktur telah dikaji. Proses ini digunakan untuk menghasilkan mikrostruktur globular untuk besi gred AISI D2. Keputusan uji kaji menunjukkan bahawa proses optimum dipilih mengikut kriteria untuk saiz ira purata dan faktor bentuk dan juga menggunakan pengagihan fasa cecair sekitar ira yang terbentuk. Dengan peningkatan suhu dan masa, fasa cecair mula meresap ke dalam sempadan ira dan tersebar lebih merata. Ira juga telah berubah ke dalam bentuk bulat, yang menjadikan saiz ira dan faktor bentuk yang lebih baik untuk kerja pembentukan-tikso. Kerja-kerja ini menunjukkan bahawa keadaan proses yang optimum untuk pembentukan-tikso D2 AISI ialah 1340°C dan masa yang diambil ialah 5 min. Ia mempunyai faktor bentuk 1.13 dan saiz zarah 51 µm.

Kata kunci: AISI D2; evolusi mikrostruktur; mengarahkan sebahagian lebur semula; pembentukan-tikso

INTRODUCTION

Semi-solid metal (SSM) processing is a relatively new technology for metal forming that was discovered at Massachusetts Institute of Technology (MIT) in the early 1970s (Spencer et al. 1972). Nowadays, the thixoforming process has wide applications in manufacturing scale because it enables high-quality production at minimal cost when compared with conventional processes. For instance, compared to casting, the quality of the final product can be improved because there are fewer defects related to solidification processes. Also, compared to forging processes, the required forming loads are much lower and thus complex shaped parts can be produced net or near-net in a one-step process (Fan 2002; Mohammed et al. 2013a). A key step for the successful operation of semi-solid processing is the appropriate production of feedstock with a thixotropic property. In general, technologies for SSM processing can be divided into two main categories based on the status of the starting material: either a liquid-phase or a solid-phase alloy (Atkinson 2005; Hirt et al. 2006; Legoretta et al. 2008). Currently, there are a number of methods that can be

used to achieve a globular microstructure, such as cooling slop, mechanical stirring, magnetohydrodynamic stirring (MHD) and direct partial re-melting (Budiman et al. 2009; Omar et al. 2011a; Spencer et al. 1972; Vivès et al. 1993). Majority of the research effort in respect of SSM processing techniques is focused on low-meltingpoint alloys such as aluminium and magnesium alloys (Arif et al. 2013; Salleh et al. 2012). In contrast, only a few studies have been undertaken on the development of high-melting-point materials for the thixoforming process. However, researchers are becoming increasingly interested in this goal due to the advantages of steel thixoforming (Mohammed et al. 2013b). In the case of high-melting-point metals, especially steel, partial re-melting is considered one of the most effective and commercially viable solid state processes to produce a non-dendritic microstructure when the metal is directly heated to temperatures between solidus and liquidus (Alfan et al. 2010; Omar et al. 2011b). This paper seeks to contribute to this stream of research by reporting the microstructural evolution of AISI D2 cold-work tool steel during partial re-melting and explaining the effects of isothermal temperature and holding times on particle size and morphology.

EXPERIMENTAL DETAILS

MATERIALS

The material used in this work is AISI D2 cold-work tool steel which was supplied after a soft annealing process, i.e. heating to 850°C, followed by cooling at 10°C/h to 650°C and finally air cooling. The chemical composition of the starting material determined by X-ray fluorescence (XRF) technique is shown in Table 1.

JMATPRO SIMULATION

JMatPro (Java-based Material Properties) is a software solution that was developed to augment thermodynamic calculations by incorporating diverse theoretical material models and a properties database that enables a quantitative calculation for the requisite material's property to be made within a larger software structure (Mohammed et al. 2012). Here, JMatPro software is used to estimate solidus and liquidus temperatures as well as the liquid fraction profile within the semi-solid zone.

DIRECT PARTIAL RE-MELTING EXPERIMENT

The direct partial re-melting experiment was performed using a vertical, high-temperature carbolite furnace with a protective atmosphere. The material was cut into samples with a size of $5 \times 10 \times 12$ mm³. When the furnace had reached the predefined temperature, the sample was lowered into the hottest place inside the furnace by using a chromel wire to ensure the rapid heating of the sample, which normally reached the predefined temperature in about 3 min. A K-type thermocouple placed inside a hole located on the $5 \times 10 \text{ mm}^2$ surface of the sample (5-6 mm deep) was used to monitor the temperature. After the desired temperature was reached, the sample was then quenched in brine water at room temperature to freeze the microstructure. The experimental process is illustrated in Figure 1.

METALLOGRAPHY AND IMAGE ANALYSIS

Microstructural characterisation was carried out using a BX-51 Olympus optical microscope and a Hitachi S3400N scanning electron microscope (SEM), equipped with energy dispersive spectroscopy (EDS). Average grain sizes were calculated by using the mean lineal intercept method (after the ASTM E112-96 standard) and the shape factor equation F=, where F is the shape factor, U is the grain circumference and A is the grain area. When F equals 1, the grains are completely globule, but when F is greater than 1, a more complex shape can be obtained (Bergsma et al. 1997). According to Hirt et al. (2009), the ideal grain size for semi-solid forming should not exceed 100 µm and globularity (shape factor) must not be greater than 2. All samples were etched using Villela reagent (1 g picric acid, 5 mL hydrochloric acid and 95 mL ethyl alcohol) to show their microstructures. The effects of temperature change and holding time on microstructure evolution were then studied thoroughly.

RESULTS AND DISCUSSION

STARTING MATERIAL

The as-received material contains surplus arrays of coarse large carbides with smaller carbides distributed homogeneously in a ferrite matrix parallel to the working direction, as shown in Figure 2. This structure has been identified in annealed cold-work tool steel and thus

TABLE 1. Chemical composition of AISI D2 (wt. %) cold-work tool steel by XRF technology

С	Si	Mn	Cr	Ni	Мо	V	Fe
1.460	0.258	0.239	11.2	0.197	0.769	0.711	Balance



FIGURE 1. Schematic of direct partial re-melting experiment process

confirms that soft annealing treatment has been carried out as described by the material supplier (Omar et al. 2009). From Figure 3, the phase stability diagrams obtained from the JMatPro simulation for as-received AISI D2 show that at room temperature, the carbides consisted of $M_{\gamma}C_{\gamma}$, MC and $M_{23}C_6$ type carbides. The initial soft annealed microstructure of the samples was observed with the SEM and was found to consist of carbides in a ferrite matrix, as shown in Figure 4. The chemical compositions of the analysed carbides and the ferrite matrix are given in Table 2. It can be seen that the M_7C_3 carbides are composed mainly of C, V, and Cr. The absence of MC carbide in the as-received AISI D2 tool steel can be explained by the soft annealing process at 850°C due to dissolution temperature of MC carbide at 740°C. The amount of $M_{23}C_6$ carbide after cooling to room temperature is negligible as compared to M₇C₃ carbides (Omar et al. 2011b).

SEMISOLID STATE MORPHOLOGY AND EVOLUTION

EFFECT OF RE-MELTING TEMPERATURES

The effect of re-melting temperature on the microstructural development of AISI D2 at the isothermal temperatures of 1270, 1300 and 1340°C with a holding time of 5 min is shown in Figure 5. It was observed that at 1270°C, grain boundaries are relatively spherical particles within a small liquid matrix that is not scattered into other parts, as shown in Figure 5(a). In the case of a re-melting temperature of 1300°C, the liquid phase starts to diffuse into the grain boundaries and the grains are separated from each other

more evenly, as shown in Figure 5(b). At 1340°C, the microstructure shows fine globule solid grains in the liquid matrix, where the grains are separated from each other because of a thick liquid phase between the grain boundaries, as shown in Figure 5(c). If enough wetting is achieved along the grain boundaries, initial shear during forming may cause the grains to flow past each other and the semi-solid slurry will flow thixotropically. The microstructure evolution proceeded with a decrement in the shape factor from 1.3 to 1.14 to give a better globular level. From Figure 6, the grain-growth effect at these temperatures was found to be in the region of 40-60 μ m.



FIGURE 3. Phase equilibrium diagram of AISI D2 tool steel obtained from JMatPro simulation



FIGURE 2. Optical micrographs of as-received AISI D2 tool steel



FIGURE 4. SEM micrograph of as-received AISI D2

TABLE 2. EDX analysis results for the carbides and ferrite matrix wt. % shown in FIGURE 4

	С	Si	Р	S	V	Cr	Mn	Fe	Ni	Mo	W	Phase
Point 1	6.03	0.1	0.08	0.03	5.07	45.95	0.34	40.58	0.07	1.46	0.29	M_7C_3
Point 2	6.70	0.16	0.02	0.24	5.52	44.51	0.49	40.62	0.09	1.57	0.10	M_7C_3
Point 3	1.61	0.27	0.02	0.02	0.3	9.2	0.35	87.20	0.29	0.29	0.45	α



FIGURE 5. Effect of holding temperature on the microstructure of AISI D2 at (a) 1270, (b) 1300 and (c) 1340°C with 5-min holding time



FIGURE 6. Differences in the primary particle size and shape factor of AISI D2 at1270, 1300 and 1340°C with 5 min holding time

EFFECT OF HOLDING TIMES

The effect of holding times of 0, 3 and 5 min on the microstructural evolution of AISI D2 during partial remelting at the isothermal temperature of 1340° C is shown in Figure 7. It was observed that at a holding time of 0 min, grain boundaries are relatively spherical particles within a small liquid matrix that is scattered into other parts, as shown in Figure 7 (a). The grains coarsen and become more globular when the holding time is increased to 3 and 5 min. This liquid phase starts to diffuse into the grain boundaries, therefore the phase scatters and spreads more evenly, as shown in Figure 7(b) and 7(c). The microstructure evolution proceeded with a decrement in the shape factor from 1.27 to 1.13 to give a better globule level. At the same time, the particle size increases from 45 to 51 µm. This shows that the grain-growth effect produces more globular

particles, as shown in Figure 8. Meanwhile, the change in grain roundness with isothermal holding time occurred in tandem with the change in grain size (19).

SEMI-SOLID MICROSTRUCTURE EVOLUTION MECHANISM

For the AISI D2 tool steel, experiments were implemented in the corresponding temperature range and the microstructure was dissected by means of classic metallography. A fine austenitic spherical primary phase was obtained after the reheating treatment of the asreceived material at temperatures of around 1340°C. A globular shape of this kind is one of the most important basic requirements for the thixoforming application. Microstructural surveillance exposed that there was a good arrangement of globular grains which are surrounded by an eutectic mixture. In this situation, the morphologies



FIGURE 7. Effect of temperature holding time on the microstructure of AISI D2 for (a) 0 min, (b) 3 min and (c) 5 min at 1340°C



FIGURE 8. Differences in the primary particle size and shape factor of AISI D2 at 1340°C for 0, 3 and 5 min

and compositions of D2 after treatment, seen in Figure 9, differ from those perspectives in the as-received samples. The transformation into spherical grains is supported by the dissolution of different types of carbides. The carbide dissolution process is beneficial in that it gives a big semisolid interval and a little temperature sensibility. However, in respect of the microstructure and temperature, it can be seen that with increasing isothermal temperature, the liquid phase starts to diffuse and distribute into grain boundaries and spread more consistently to make a solidphase spherical structure with favourable grain size for thixoforming (Mohammed et al. 2013c). Figure 9 shows the EDS analysis results from the SEM image taken from points 1 and 2. From point 1, the EDS results confirm that metastable austenite was present, particularly in the globular grains. The residual interspaces were filled by precipitated eutectic carbides on the grain boundaries

and on the lamellar network which is indicated by the increased content of chromium (Cr), vanadium (V), and carbon (C) in the eutectic area, as shown at point 2.

CONCLUSION

The microstructural evolution of AISI D2 cold-work tool steel has been examined at various isothermal temperatures and holding times during partial re-melting. Microstructural observation showed that at low isothermal temperature and short holding time, discontinuous and non-penetrated grain boundaries occurred. With increasing isothermal temperature and holding time, the liquid phase starts to diffuse into the grain boundaries and spreads more evenly, which makes the grain size and shape factor more favourable for thixoforming. The present work shows that the optimal process condition for the thixoforming of AISI



FIGURE 9. SEM micrographs of AISI D2 sample after being partial re-melted at 1340°C with EDX analysis at points 1 and 2. Note that (b) is the area within the white rectangle shown in (a)

D2 is 1340°C and 5 min holding time, having a shape factor of 1.13 and a particle size of 51 μ m.

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