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Solid–liquid structural break-up in M2 tool steel for semi-solid metal processing

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Abstract The success of semi-solid metal processing mostly depends on the formation of suitable starting microstructure, which must consist of solid metal spheroids in a liquid matrix. Various methods of obtaining this structure have been established; they include recrystallisation and partial melting (RAP), strain-induced melt-activated (SIMA), or simple mechanical stirring, to name a few. These methods, as widely discussed, have mostly been applied with light alloys, mainly aluminium based. This article discusses solid–liquid structural break-up in M2 tool steel subjected to a direct re-melting procedure from the as-annealed condition. The role of carbide dissolution in the grain boundary liquation of the steel is described. This leads to the production of near spheroidal solid grains in a liquid matrix, a microstructure suitable for the thixoforming process. Microstructural examination revealed that carbide particles contained in bands at 1220 °C slowly disappeared with temperature. At 1300 °C, the solid grains seemed to be free from carbides. Most of the carbides had now re-precipitated at the grain boundaries. Thixoforming carried out at 1340 and 1360 °C revealed the thixotropic properties of the semi-solid metal slurries. The results indicate a widening of the range of potential routes to thixoformable microstructures.

Introduction

The initial work that led to the interest in semi-solid metal processing (SSM) can be traced back to studies by researchers at The Massachusetts Institute of Technology in the early 1970s [1]. This work was originally directed at the problem of hot tearing in alloy castings but it was later realised that a new technology for near-net shaping of complex shapes had been discovered. This SSM technology can be generally defined as a forming process that shapes metal components in their semi-solid state [2–4]. There are several routes for SSM processing including thixoforming where material with an appropriate microstructure is initially solid and is reheated into the semi-solid state for forming.

There is a variety of routes to obtain the appropriate microstructure for thixoforming i.e. a microstructure which consists of solid spheroids in a liquid matrix once the material is reheated into the semisolid state. Some are based on the liquid state (e.g. see [5]). The more established solid-state routes are recrystallisation and partial melting (RAP) and strain-induced melt-activated (SIMA). The first involves cold or warm working below the recrystallisation temperature followed by heating above the solidus of the material, while the latter involves hot working between recrystallisation and solidus temperatures, followed by cold working so as to produce a critical level of strain, before the material is reheated above the solidus.

Now, the process is mainly established in the production of parts made of aluminium alloys. Work in the

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thixoforming of high-temperature materials, such as steel, is still at its initial stage; this is mainly due to the high processing temperatures involved [6–9]. For thixoforming to be possible, it is preferable for an alloy to have an appreciable melting range and, before forming, the microstructure must consist of solid metal spheroids in a liquid matrix. Here, a commercially produced M2 tool steel alloy, i.e. being hot worked (by GFM), tempered and annealed, was directly reheated in a protective atmosphere from room temperature to above its solidus with no prior additional cold working. GFM (from the German words for *Gesellschaft für Fertigungstechnik und Mashinenbau*) forging is based on the radial forging principle whereby a long workpiece is hammered by four forging tools.

One major attraction of thixoforming high-temperature materials (e.g. steels) is the low forging force involved during thixoforming as compared to that in conventional forgings [6]. This means that more intricate and complex shapes can be formed faster with some reduction in forming steps and with near-net shaping capabilities [6, 10–12]. Other major advantages include prolonged die life due to decreased thermal shock (forging below liquidus as against castings), weight savings in components with less porosity than conventionally, plus improved usage of feedstock materials because of improved designs.

This article describes the microstructural development of an M2 tool steel when directly reheated into its semi-solid zone from the as-annealed condition. In particular, it discusses the role of carbide dissolution in the grain boundary liquation of the steel, hence producing near-spheroidal solid grains in a liquid matrix, a microstructure suitable for the process.

Experimental procedure

The M2 high-speed steel used was produced by GFM hot forging at a temperature approximately 1150 °C. It was then tempered at 650–750 °C for about 4 h, and subsequently annealed for 8–10 h at 860 °C before being furnace cooled to ambient temperature. This is the state in which it is conventionally received from the supplier (Barworth Flockton Limited, UK). All the heat treatment was carried out in a controlled atmosphere to prevent oxidation and de-carburisation. The chemical composition of the starting material is given in Table 1.

Table 1 Chemical composition of the M2 tool steel compared to the nominal

M2	Chemical composition (wt%)[a]								
	C	W	Mo	Cr	V	Co	Si	Mn	Ni
Nominal	0.85	6	5	4	2	–	–	–	–
Chemical analysis	0.87	6.20	4.68	3.98	1.72	0.48	0.07	0.04	0.02

[a] Balance is Fe

Partial remelting was carried out using a vertical, high-temperature quench Carbolite tube furnace, capable of reaching a maximum temperature of 1500 °C. The as-received billet was cut into coupons of approximately 5 × 10 × 12 mm^3. A K-type thermocouple was placed inside a hole located on the 5 × 10 mm^2 surface of the coupon (5–6 mm deep), to ensure that the sample had reached the predefined quenching temperature, and hung inside the furnace. The as-received samples were subjected to different holding temperatures and times (it takes about 4 min for each sample to reach the respective targeted temperatures). They were quenched in a salt bath after the predetermined experimental parameters had been established to freeze the structures. To ensure that the sample is cooled rapidly, its dimensions must be kept to a minimum while at the same time having enough strength in the mushy state to provide the presence of thick walls around the thermocouple inside the thermocouple hole. A coupon thickness of 5 mm was found suitable for the purpose. The selected temperatures were from 1220 to 1360 °C at 20 °C intervals and for various holding times. The heating was carried out in an argon atmosphere to reduce oxidation. The quench furnace set-up is shown in Fig. 1.

The microstructural characterisation was carried out using KS-400 Imaging System Release 3.0 software

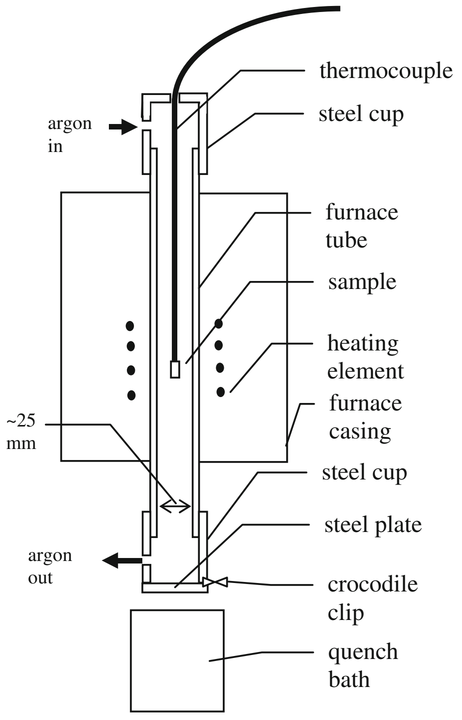


Fig. 1 Schematic of the furnace set-up for remelting experiment

connected to a Reichert-Jung Polyvar MET optical microscope and Jeol JSM 6400 scanning electron microscope. The grain or cell size and the volume fraction of liquid were, respectively, measured adopting the Mean Lineal Intercept method as outlined in ASTM E112-96 standard [13] and by the Systematic Manual Point Count as outlined in ASTM E562-99 standard [14]. All samples were etched using 5% Nital (5 mL $HNO_3 + 95$ mL methanol or ethanol).

Results and discussion

Figure 2a and b is the scanning electron micrograph under back-scattered electron imaging mode for the as-received M2 tool steel. Figure 2a shows carbide particles in the ferrite matrix contained in bands parallel to the working direction along the as-received billet. Such banding is typical of good manufacturing practice for most commercially produced high-speed steels [15]. Figure 2b shows two different types of carbides present in the as-received M2, termed here the ‘whitish’ (W) and ‘greyish’ (G) carbides. Energy dispersive spectroscopy (EDS) analyses showed that the former are rich in tungsten and molybdenum (an M_6C type carbide), while the latter are vanadium–tungsten–molybdenum-rich MC-type carbides. Table 2 shows the chemical composition (wt%) of these two types of carbides. The absence of $M_{23}C_6$ chromium-rich carbide can be explained by the fact that the as-received M2 in this work was hot forged at 1150 °C, above the dissolution temperature for these carbides [15].

When directly re-heated from its as-annealed (as-supplied) form to 1340 and 1360 °C, M2 tool steel exhibits the appearance of a conventional thixoformable microstructure of spheroidal solid phase within a liquid matrix [16] (see Fig. 3). Depending on the holding times, the average grain size at 1340 °C is between 30 and 43 µm, whereas at 1360 °C, the average grain size is between 31 and 48 µm. Also the liquid content at 1340 and 1360°C, as calculated from quenched samples, is within the 20–50% range normally carried out in thixoforming.

Table 2 The characteristic chemical compositions of the whitish and greyish carbides as shown in Fig. 2

Carbide	Wt%					
	W	Mo	Cr	V	Fe	Co
Whitish	29.1	27.0	4.4	3.6	35.7	0.3
	29.0	26.1	4.5	3.8	36.0	0.5
	29.1	26.1	4.8	3.6	36.1	0.3
Greyish	12.2	13.7	4.0	47.4	20.4	–
	14.7	14.0	4.3	44.7	21.9	0.3
	15.9	13.9	4.7	39.1	26.4	–

The relatively fine grain size is thought to be related to carbide pinning of grain boundaries. The huge amounts of carbides are responsible for retarding the grain growth in tool steels; the strongest pinning effect is from vanadium-rich carbides [15]. The vanadium-rich and the tungsten–molybdenum-rich carbides, as shown in Fig. 2, restrict the grain growth during the ferrite-to-austenite phase change of M2 (which takes place around 800 °C). Note that carbide particles (in this case, cementite) are also responsible for controlling the rate of growth of ferritic grains in alloy steels, by pinning the boundaries [17].

The influence of carbides on the development of spheroidal structures is shown by the microstructural development sequence from the sub-solidus temperature of 1220 °C and various other temperatures above the solidus (around 1240 °C) up to 1300 °C (see Fig. 4). For this purpose, a holding time of 4 min was applied in order to achieve as near an equilibrium condition as possible. The microstructures show that below the solidus, the majority of the carbide particles are still contained in bands parallel to the working direction, a characteristic that is also observed in the as-supplied material. Very fine grains are seen in the background in the matrix (see Fig. 4a). However, as the temperature rises, most of the original carbide particles seem to have been dissolved and new ones are precipitated at the grain boundaries (i.e. carbon and other alloying elements diffuse to the grain boundaries, resulting

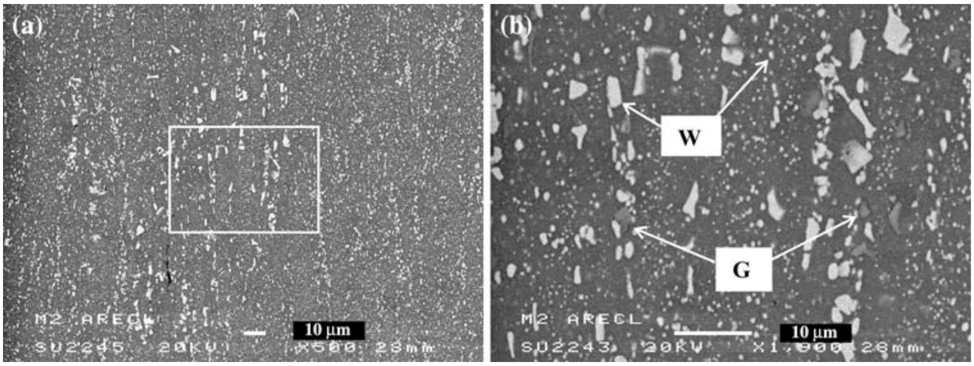


Fig. 2 SEM (backscattered) micrographs of as-received M2 showing carbide segregation caused by forging. The area outlined in (**a**) is equivalent to (**b**) carbides that are distinguished as either whitish (*W*) or greyish (*G*) (longitudinal direction is in the vertical direction)

Fig. 3 Optical micrographs of GFM M2 at **a** 1340 °C and **b** 1360 °C at 0 min holding [14]

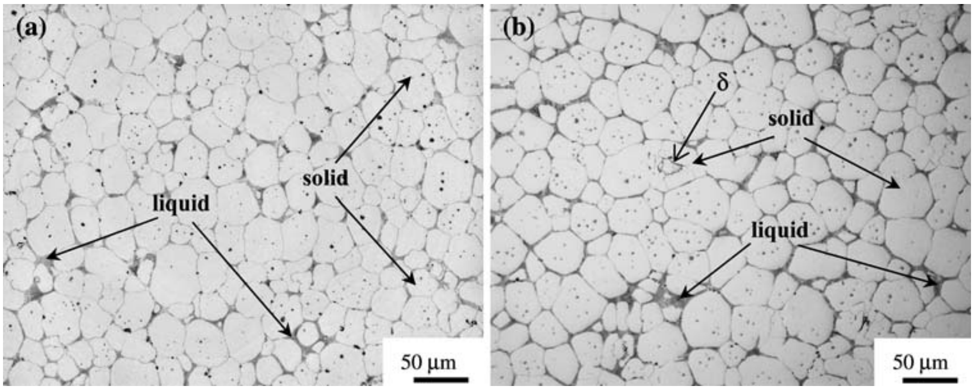
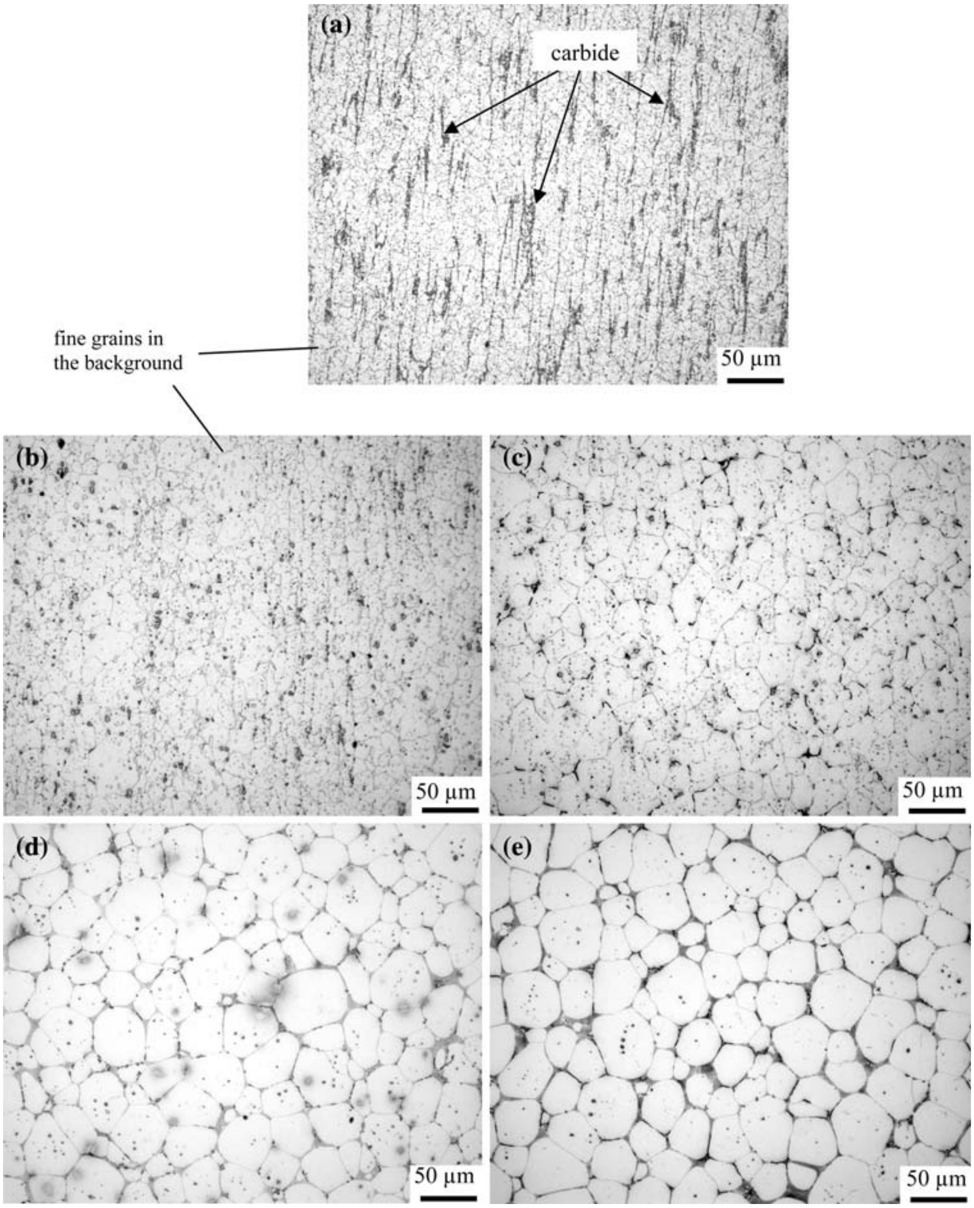


Fig. 4 Optical micrographs of as-supplied M2, isothermally re-heated in a controlled atmosphere for 4 min holding time at **a** 1220, **b** 1240, **c** 1260, **d** 1280, and **e** 1300 °C



in the re-formation of new carbides during subsequent cooling). At 1300 °C, the now roundish austenitic grains are seen to be free from carbides.

Figure 5a and b is the SEM micrograph in secondary electron mode showing examples of carbide morphology at 1340 °C (4 min holding time) at different magnifications.

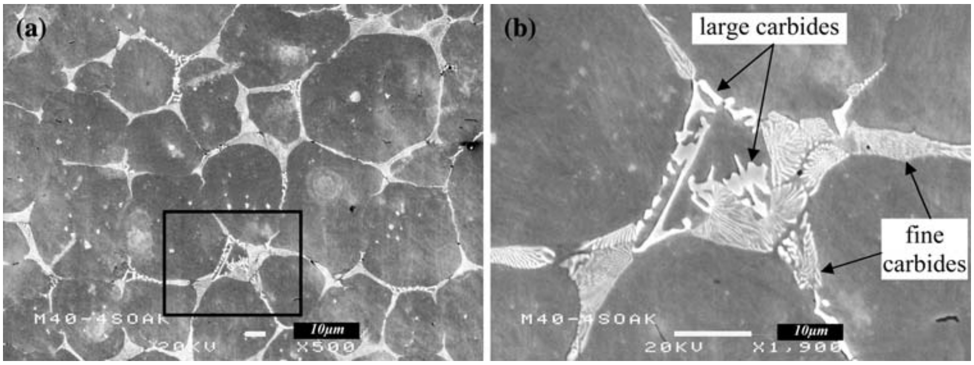


Fig. 5 SEM micrographs showing examples of carbide morphology at 1340 °C (4 min holding time) at different magnifications, in secondary electron mode. **b** is the area within the *black rectangle* shown in (**a**)

EDS analyses showed that the large carbides are rich in vanadium, tungsten and molybdenum, which could be variants of the MC-type carbide. The finer carbides are tungsten–molybdenum-rich M_6C-type carbides.

It is observed that M2 readily produces near-spheroidal solid grains when heated into its semi-solid zone. As stated in the literature, certain quantities of eutectic carbides will dissolve first when certain steels are heated towards the melting point [15, 18, 19]. Here, the high volume fraction of eutectic liquid will penetrate into grain boundaries during the liquation process. Subsequently, melting at sharp asperities and solidification in regions of negative curvature due to diffusion processes follows, resulting in a morphology of nearly rounded solid particles within a liquid matrix.

The results obtained thus far show that M2 can be directly re-heated to its semi-solid range from the as-received state without having to go through the conventional feedstock preparation routes, like RAP, SIMA, and various others, as summarised in Fig. 6. This indicates a widening of the range of potential routes to thixoformable microstructures.

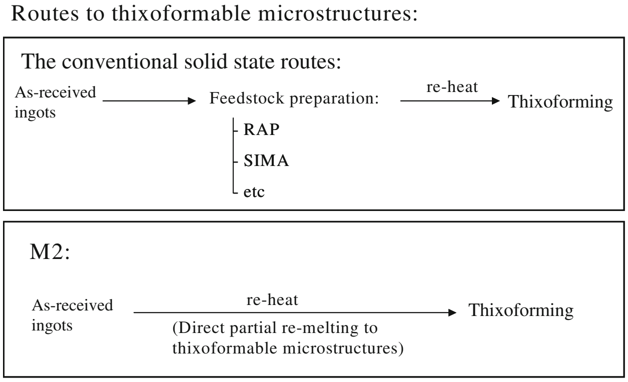


Fig. 6 The different routes to obtain suitable thixformable microstructures

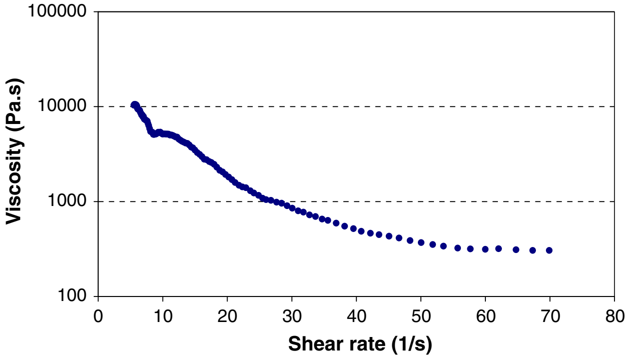


Fig. 7 Viscosity-shear rate of M2 when thixoformed at 1360 °C

An illustration of the thixotropic property of the slurry obtained during the compression of semi-solid slug at 1360 °C is shown in Fig. 7. The detailed set-up of the compression experiment and the corresponding load–displacement signals can be obtained by reference to [16]. Data from the load–displacement signals were used to estimate the viscosity of the semi-solid slurry following the method of Dienes and Klemm [20] in solving Stefan's equation for flow between two parallel planes. The compression force F is:

$$F = -\frac{3\mu v^2}{2\pi h^5}\left(\frac{\mathrm{d}h}{\mathrm{d}t}\right) \qquad (1)$$

where μ is the viscosity of the slurry, v is the volume of the semi-solid slug, h is the instantaneous height of slug under compression, and t is time. The average shear rate, $\dot{\gamma}_{\mathrm{avg}}$, at any instant during compression can be obtained from:

$$\dot{\gamma}_{\mathrm{avg}} = -\frac{R}{2h^2}\left(\frac{\mathrm{d}h}{\mathrm{d}t}\right) \qquad (2)$$

where R is the radius of the specimen at time t.

Viscosity is shown to be a strong function of shear rate, i.e. decreasing with increasing shear rate. In rheological terms, the slurries are said to exhibit pseudoplasticity, by showing shear-thinning behaviour. This behaviour is also shown when processing at 1340 °C [16]. Figure 7 and the thixoforming behaviour are evidence that the near-spheroidal microstructure typified in Fig. 4e is suitable for semi-solid processing.

Conclusion

The microstructures and phases of as-annealed M2 tool steel in the as-received condition and within the semi-solid state have been studied. The as-received material shows carbide particles contained in bands parallel to the working direction of the as-received billet. At sub-solidus temperature of 1220 °C, these bands are still clearly seen. When directly reheated from the as-received condition into the semi-solid zone, the grain boundary carbides are dissolved. As a result, liquation of grain boundaries occurred and the material exhibits fine equiaxed solid grains that are surrounded by liquid matrix, indicating a widening of range of potential routes to thixoformable microstructures. This route to a thixoformable microstructure has not been identified previously for light alloys which are the staple of the semi-solid processing industry. High-temperature alloys present particular challenges for the preparation of suitable feedstock and this provides an useful additional and economical route. Thixoforming carried out at 1340 and 1360 °C revealed shear-thinning behaviour of the semi-solid metal slurries and hence confirmed the suitability of the route.

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