Thixoforming of a high performance HP9/4/30 steel

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Abstract

Thixoforming is a forming process that shapes metal components in their semisolid state. Prior to forming, the microstructure of the alloy consists preferably of solid metal spheroids in a liquid matrix, which can be difficult to achieve with hot-worked, high alloy steels prone to strong microsegregation bands. A high performance HP9/4/30 steel has been assessed for thixoformability through a direct remelting route. Partial remelting was carried out between 1430 and 1470°C. Liquation occurred initially at grain boundaries, then also along the segregation bands. With increasing time and hold temperature, these "columns" broke down into shorter, more equiaxed segments, offering more chance of being thixoformed. Successful thixoforming producing net-shape demonstrator parts was achieved at processing temperatures in the range of 1470 – 1480°C, which corresponds to approximately 50 to 80% liquid (based on Differential Thermal Analysis). A thin solid skin, formed on the surface of the slug as a result of heat loss, had prevented the slug from collapsing. The resulting thixoformed products are discussed in relation to their load-displacement signals.

Keywords: Thixoforming; Microsegregation; Direct remelting route; Partial remelting

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1. Introduction

Thixoforming is a forming process that shapes metal components in their semisolid state. Prior to forming, the microstructure of the alloy consists preferably of solid metal spheroids in a liquid matrix, which can be difficult to achieve with hot-worked, high alloy steels prone to strong segregation bands. Alloy slurry with a non-dendritic microstructure is said to be in a thixotropic state (from the Greek words of *thixis* which means 'the act of handling' and *trope* meaning 'change'), whereby the internal structure of the material is changed by an external load. If an alloy in this state is sheared, this will result in a fall in its viscosity and it will flow like a liquid, but if allowed to stand it will thicken again. The behaviour of this type of slurry, which is acting like a non-Newtonian fluid, was first discovered by Spencer et al [1] and has since led to extensive work on the thixotropy of alloy slurries [e.g. reference 2].

On thixoforming of high temperature materials, one major attraction of thixoforming such materials, e.g. steels, is the low forging force involved during thixoforming as compared to that in conventional forgings [3]. This means that more intricate and complex shapes can be formed faster with some reduction in forming steps and with near net shaping capabilities [3-6]. Other major advantages include prolonged die life due to less 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 [7-8].

The work described in this paper is the result of development in the semi-solid processing of a high melting point alloy: a high performance HP9/4/30 steel having a

typically banded starting microstructure. The development of its microstructure in the semi-solid state is discussed. The thixoforming induction heating trials and the resulting load-displacement signals of the thixoformed products are explained.

2. Experimental procedure

2.1 Material

The high performance HP9/4/30 steel used was produced by a vacuum arc remelting (VAR) process and rolled at 1250-1310°C. The high temperature of rolling is to achieve homogenisation in the ingot. It was subsequently normalised at 899-927°C (air cooling to room temperature), hardened by heating to 843°C (oil or water quenched) and refrigerated at -73°C (warming in air to room temperature), double tempered at 538°C and lastly double annealed at 677°C (air cooling) and 621°C (air cooling), according to an AMS 6526C standard [9]. Its chemical composition is compared to the standard in Table 1.

2.2 Differential Thermal Analysis (DTA)

DTA analysis was carried out to estimate the solidus, liquidus and liquid fractions within the semi-solid zone of the supplied material. The alloy was cut into small pieces of 70-90mg weight for DTA tests using a Perkin Elmer Differential Thermal Analyser DTA7. Alumina of about 100mg weight was used as the reference material. The heating rate employed was 20°C per minute. The heating was carried out in an argon

atmosphere to prevent oxidation. Some examples of DTA-measurements on determination of the solidus, liquidus and liquid fractions within the semi-solid zones of a few steel grades can be found in [10].

2.3 Partial remelting experiments

The partial remelting experiments were carried out in order to gain some insight into the microstructural development of the starting material when in the semi solid state. These can be used to estimate relationships between microstructure, cell size and shape, and holding time at a given temperature of the material; hence to establish a suitable condition for thixoforming. Here, a direct remelting route was employed, that is, partial remelting was carried out straight from the as-received condition. The as-received billet was cut into coupons of approximately 5x10x12mm. The tests were carried out using a vertical high temperature quench Carbolite tube furnace, capable of reaching a maximum temperature of 1500°C. The set-up is shown in Fig. 1. An R-type thermocouple was placed inside a hole located on the 5x10mm surface of the coupon (5-6mm deep), to ensure that the sample had reached the predefined quenching temperature, and hung inside the furnace. After the predetermined holding time, the sample was immersed into the quencher. 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 [11] to provide the presence of thick walls around the thermocouple inside the thermocouple hole. A coupon thickness of 5mm was found suitable for the purpose. The selected temperatures were 1430, 1450, 1460 and 1470°C (within the semi solid zone – based on the DTA) at various holding times of 2, 4, 8 and 16 minutes. The heating was carried out in an argon atmosphere to reduce oxidation as this is one of

the major issues with thixoforming of steel at high temperatures. The use of protective atmospheres has also been adopted by other authors in order to avoid excessive high temperature oxidation [12,13].

2.4 Image analysis

The microstructural characterisation was carried out using KS-400 Imaging System Release 3.0 software connected to a Reichert-Jung Polyvar MET optical microscope. The grain or cell size was measured adopting the Mean Lineal Intercept method as outlined in ASTM E112-96 standard [14] while volume estimation of liquid fraction was carried out adopting automatic image analysis as outlined in ASTM E1245-95 standard [15] using ImageJ 1.28u, a commercially available image analysis software package. Unless otherwise stated, all samples were etched using 5% Nital (5ml HNO_3 + 95ml methanol or ethanol).

2.5 Thixoforming heating trials

Thixoforming heating trials were carried out in order to determine the conditions required for a successful thixoforming. A slug of 40mm diameter and 40mm length was used and the heating was by means of an induction system having a current frequency of 1000Hz and a maximum power capacity of 120kW, with the induction coil having a diameter of 130mm. The trials were carried out using the thixoforming press at the University of Sheffield, Sheffield, United Kingdom. The schematic diagram of the press is shown in Fig. 2. The slug was placed on a non-metallic extension (pedestal) on the ram in the thixoformer and raised to the heating position in the centre of the coil.

The top and bottom surfaces of the slug were both insulated with a layer of Kaowool pad of approximately 2mm thick. Kapranos et al. [16] had shown that this procedure reduced heat losses and gave a more uniform temperature distribution in the slug during heating. Temperature profiles during the heating trials were monitored using R-type thermocouples which were inserted right at the centre and near the edge of the slug as shown in Fig.3. Various heating sequences were experimented with. A three step heating sequence of 24kW for 6 minutes, 96kW (1 ½ minutes), and 91.2kW (2 minutes) was finally adopted prior to forming. The temperature-time-capacity profiles of the heating cycle are shown in Fig. 4 (a).

There are several issues associated with temperature measurement and control during the heating trials. The first is the accuracy of the temperature measurement; this is ±1°C. The second issue is control of temperature around the identified process temperature. This proved to be quite challenging with this material and the high temperatures involved. It will be shown in the Results and Discussion section that, although the desired process temperature was 1470°C, the temperature, at half a minute or longer soak times, actually varied between 1470 and 1480°C. This was related to radiation losses experienced by the slug and is discussed in section 3.

The third issue is the reproducibility of a temperature profile given an identical heating schedule. Fig.4 (b) shows two runs with identical heating schedules. The slopes during the heating up stages are almost identical. The range (1470 - 1480°C) on the plateau is also identical. Reproducibility at the plateau stage is linked with the second issue.

2.6 Thixoforming

Thixoforming was carried out using the press, insulating pads and slugs of identical size to that mentioned above. The press has the capacity of producing a 100kN load during forging and is able to transfer a slug of material to be injected into a die at a maximum velocity of 1000mm/s. It is controlled by a computer equipped with a Servotest DCS 2000 digital control system. The data collection acquisition rate is up to 2kHz. Load-displacement signals during forging were obtained from a pressure transducer sensing hydraulic fluid pressures exerted by the ram. The signals obtained are collected by the computer controller and easily transferred into Microsoft Windows Excel software for analysis. For the monitoring of temperature, the measurement of the temperature with thermocouples could not be easily integrated in the gas chamber during thixoforming. As a result, instead of depending on the temperature-capacity, subsequent thixoformings were carried out based on time-capacity of the induction heating schedule established in the heating trials.

For the selection of the die materials for thixoforming of steels, it is known that conventional hot working tool steel dies may soften at temperatures of around 600°C [17]. Work on thixoforming of high temperature materials had been carried out using various die materials and concepts, including graphite, ceramics (e.g. sindanyo and alumina) and composite die concepts consisting of multiple materials [5,17,18]. Graphite dies were used in this work because they can be easily and cheaply prepared. Furthermore, they had already been shown to be successful in thixoforming of some high temperature materials [19]. The dies were housed in a split steel die box and

clamped at the top of the press. They produced finger-like thixoforgings of 60 mm length and 10 mm by 18 mm section, which could be machined into specimens for tensile testing. The slug was heated in an argon gas environment to reduce oxidation. The optimisation was carried out in terms of die filling velocity (of 350-1000mm/s), dwell time (90-180s) (at a final load of 100kN) and hold time (0-2 minutes) at processing temperatures of between 1470 and 1480°C. In this paper, a forging carried out immediately when the slug has just reached 1470°C is considered as a 'thixoforming at 1470°C at zero minute soak'. Forgings carried out after a hold of 0.5, 1 and 2 minutes at a 'process temperature' of 1470°C (which, in practice, meant in the range 1470 – 1480°C, see earlier) are described as 'thixoforming in the range 1470-1480°C at 0.5, 1 and 2 minutes' respectively.

3. Results and discussion

3.1 Starting material

An optical micrograph of the as-received high performance HP9/4/30 steel is shown in Fig. 5. The ingot had been subjected to rolling at 1250-1310°C. The high temperature was to assist in achieving some homogenisation in the ingot. A complex heat treatment process had also been applied, that is, normalising, hardening, refrigerating, double tempering and annealing at temperatures and conditions as mentioned in section 2.1. The complex microstructure shown by the material is related to this complex heat treatment history. The longitudinal surface (shown in Fig. 5) shows some evidence of banding along the longitudinal direction of the ingot. The

banding originates from the microsegregation in the primary dendritic structures of the original ingot during the VAR process [20]. The rolling has then led to directionality in this microsegregation. Homogenisation and the heat treatment process involved will have reduced the microsegregation effect but the alloy contains high levels of slowly diffusing elements and therefore full homogenisation is impractical. The longitudinal banding observed is typical for these materials in normal commercial practice and conforms to the acceptable standard in the longitudinal banding examination mentioned in an EMS96247 standard [21]. The microstructure of the starting material consists of a mixture of lath and tempered martensites, and a fraction of retained austenite in martensitic matrix [22-24]. The grain size is predominantly 60-80μm with occasional grains of about 120μm (revealed when etched with Vilella's reagent), which conform to the AMS6526C standard [9].

3.2 Microstructural development during partial remelting.

Prior to the partial remelting experiments, DTA analysis was carried out to estimate the solidus, liquidus and liquid fractions within the semi solid zone of the supplied materials using the set-up mentioned above. The DTA results are shown in Fig. 6. The liquid fraction curve indicated a steep increase in liquid fraction values between 1460 and 1480°C. The solidus is estimated at 1425°C and the liquidus at 1500°C, while 20% and 50% liquid correspond to 1455°C and 1470°C respectively. Note that thixoforming is normally carried out at fraction liquid between 20-50% [7,25,26]. The estimation of the solidus and liquidus temperatures and also the liquid fraction at various temperatures in the semi solid zone, is helpful in determining the temperatures for the isothermal re-heating tests in the semi solid zone. The quenched coupons from the

remelting tests could be used to assess further the liquid fraction against temperature. Fraction liquids at different temperatures at 16 minutes holding time from image analysis are also shown in Fig. 6. The liquid fraction underestimation by the image analysis as compared to the DTA is due to insufficient cooling rate to instantaneously freeze the microstructure, resulting in further solidification of liquid on the solid phase.

Fig. 7 is a micrograph from the coupon that was heat treated to 1430°C and held for 2 minutes. Representative optical micrographs of the microstructural evolution with increasing temperatures and holding times are shown in Figures 8 – 10. Only the micrographs at 2 and 8 minutes holding times are shown here.

The microstructures at 1430°C (i.e. at a temperature just above solidus) show some distinct polygonal grains. Thin liquid films are seen on grain boundaries but no liquation is obvious at the microsegregation bands. The size of these polygonal cells at 2 minutes holding is 455 ± 40 μm. At 1450°C, the polygonal structure is less obvious, i.e. sharp edges and straight lines of the grain boundaries have diminished. Liquation is seen not only on grain boundaries but also on microsegregation bands. The grain size at 2 minutes holding is 338 ± 40 μm. This is about two thirds of the size at 1430°C at 2 minutes holding. The reduction in size could be the result of the liquated bands (which are absent at 1430°C), leading to some smaller truncated grain structures at 1450°C. At 1460°C, major liquation is seen to occur at the bands rather than at grain boundaries. These liquated bands are again seen at certain places (e.g. in Fig. 9 (b)), breaking up the primary cells producing smaller truncated grains. Grains from re-solidified liquid during the quench are also seen particularly within bigger bands such as in Fig. 9 (a). Because of the non-homogeneous nature of the bands, a homogeneous grain structure

could not be achieved across the material. Similar observation is seen at 1470°C as at 1460°C. Major liquation occurs at the microsegregation bands, and these bands when seen in transverse section could form interconnected networks with each other and with entrapped liquid pools within the grains to produce smaller grains. A great amount of grains from the re-solidified liquid are also obvious within thick bands particularly after 8 minutes holding. Apart from these grains, the grain size at 8 minutes holding could be as low as 120μm (compare with the size at 2 minutes holding of 318 ± 32 μm); the bands now appear to have disintegrated.

In general, the thin liquid films formed at grain boundaries thickened as time and temperature increased. However, at 1450 and 1460°C, a substantial amount of these grain boundaries still exhibit very thin liquid films that might be insufficient for grains to flow past each other. At 1470°C, particularly after 8 minutes holding, thick liquid films are achieved at grain boundaries (plus the formation of grains from re-solidified liquid). These microstructures could be suitable for the thixoforming process (despite their lack of conventional appearance) as the initial shear may cause the liquid pools to link up [27]. Detailed discussion on the microstructural development of the material during partial remelting from two different starting conditions can be found in [28].

3.3 Thixoforming

From the results of the partial remelting experiment, a suitable temperature for thixoforming the HP9/4/30 appears to be at 1470°C (Fig. 10 (b,c)). Hence, heating trials were carried out to determine suitable heating conditions (power sequence and time) of the thixoformer induction furnace to reach that temperature. The slug was

heated by means of an induction system and, for reasons of productivity and material characteristics, was heated as quickly and as uniformly as possible [3,25]. Various power settings were experimented with and it was found that a high power of 24kW (at 1000Hz current frequency) gave initial relatively rapid heating of the slug while maintaining its position inside the induction coil. The thixoforming press set-up at Sheffield only allows a slug to be rested on a pedestal without a holding mechanism, hence an excessive power input at the initial stage of heating could cause the steel slug to jump or be displaced due to the sudden strong electromagnetic field produced by the induction system.

The thixoforming heating cycle shown in Fig. 4 indicates that the power capacity was initially at 24kW with a rapid rise of temperature (~5°C/s) until a plateau was reached at about 780°C. Here the material has reached its Curie point, above which it ceases to be magnetic. The power then was raised sharply to 96kW with a temperature rise of about 7°C/s, hence quickly reaching 1400°C in about 1½ minutes. This was followed by a fusion and homogenising third stage, whereby the power was slightly reduced to 91.2kW resulting in a slower and relatively uniform heating at very high temperatures of about 1470-1480°C. The heating profile also showed that the surface of the slug heats up first due to the 'skin effect' phenomenon [15,29,30]. The inside of the slug heats up more slowly via thermal conduction from the hotter surface. At the end of the heating period, the inside of the slug is hotter owing to the radiation losses from the outer surface. The material exhibits a small semi-solid range of about 70°C (i.e. ~1430 – 1500°C) (see Fig. 6) with a rapid increase in liquid fraction from about 30% to 80% from 1460 to 1480°C. The radiation losses at these high temperatures had produced a thin solid skin surface which was easily broken during forging. The effect could be of

an advantage for the thixoforming process; in that it reduces the possibility of the billet collapsing, particularly if as high as 50 to 80% liquid is involved (as in this particular case with HP9/4/30) at forging.

Although the partial remelting tests on small coupons indicated that a suitable structure for thixoforming could be achieved at processing temperature of 1470°C at 8 minutes holding, it was found in practice that it was almost impossible to maintain a constant temperature within the slug at this extremely high temperature for such a period due to the radiation losses as mentioned above. The heating profile showed that the fusion stage achieved was within the range of 1470-1480°C. The maximum holding time used during the thixoforming tests was 2 minutes.

Fig. 11 shows four thixoformed fingers forged at 500mm/s ram speed (die filling velocity), 90s dwell, at temperatures of 1470°C at zero minute hold and 'in the range 1470-1480°C' at 0.5, 1 and 2 minutes holding times, from a cylindrical slug of 40mm diameter by 40mm height. They showed that incomplete filling was obtained at the zero and 0.5 minutes hold, while at 1 and 2 minutes, complete filling was achieved with the fingers showing the exact shape and surface detail of the die cavities. Fig. 12 shows the load-displacement signal profiles of the thixoformed fingers shown in Fig. 11. The profiles show only part of the stroke length of the ram just after the slug first impacts the top of the die denoted by the zero position on the ram displacement axis (note that the height of the slug is 40mm and that the ram movement is to the left of the profiles). Generally all the profiles exhibited a similar pattern and can be divided into four distinct zones. As an example, these zones are marked on the 'zero minute soak' profile of Fig. 12: the slug prior to it first touches the top die, zone **(1)**; it showed a peak load **(2)** just

after the slug first touches the top die; followed by a significant drop in load thereafter **(3)** and a rapid increase of load when the material fills the die **(4)**, or in the case of incomplete filling, freezes prematurely. However, an increase in processing temperature and holding time showed a decrease in the first peak load encountered.

Load versus displacement curves under an actual thixoforming process are discussed here as against load-displacement curves from rapid compression tests (on aluminium alloys) as reported by other researchers such as Hogg et al. [31], Chayong [32] and Liu et al. [33]. They carried out rapid compression tests with instrumented dies in order to simulate the thixotropic flow behaviour of materials during the rapid compression experienced by the billet during an actual thixoforming condition. Kopp et al. [34] on the other hand, had studied the thixotropic behaviour of a Sn-15%Pb alloy by plotting load-displacement profiles from simple compression tests. Both sets of compression tests showed a similar pattern of load-displacement profiles to the ones shown here. The four zones of load against displacement profiles shown in this work could then be explained as: **(1)** near zero load zone prior to reaching the die. The load is not equal to zero prior to contact with the die because in this work the load signal was collected from the ram, as against the load cell at the back of the die in Liu et al. [33], Hogg et al. [31] and Chayong's [32] work. Since the load signal was converted by a pressure transducer from the hydraulic fluid pressure exerted by the ram, the movement of the ram in the majority of the stroke length will give non-zero load values prior to first impact of the billet with the die [35]. **(2)** Structure breakdown zone. The peak load should be the initial structure breakdown load, which represents the load required for de-cohesion of a proportion of insufficiently wetted grain boundaries and segregation bands. The load required to overcome the resistance to shear of the broken down

structure could also contribute to the sharp peak [31]. The magnitude of the peaks could be an indicator of the amount of insufficiently wetted grain boundaries and segregation bands present at the processing condition. The peak load of about 17kN at zero minute holding is more than three times the peak load at 2 minutes holding. **(3)** Thixotropic zone. Once the breakdown phenomenon is completed, the solid grains are now surrounded by a liquid phase and so, sufficient grain boundary decohesion is achieved and the semi-solid material starts to show thixotropic flow behaviour (or only partially for the case of the zero and 0.5 minute holding time). A flow load as low as 1kN was achieved when processing at 2 minutes holding. **(4)** Rapid increase in the load as the semi-solid slurry fills the die cavity or freezes (resulting in incomplete die filling). Also note that this load for the 0.5 minute holding time rises more rapidly compared with the 1 minute holding although the structure breakdown zone peaks are about the same magnitude. The thixoforming load-displacement signals obtained indicate that, to achieve complete die filling, it would be desirable to have a low structure breakdown load in zone (2), and an almost constant or very slow rise of thixotropic flow load of zone (3). The work so far shows that these load signals are greatly influenced by processing temperatures and holding times.

4 Conclusions

The work carried out has demonstrated the feasibility of thixoforming a high performance, high alloy HP9/4/30 steel with typically microsegregated starting structures. The material is successfully thixoformed from its semi-finished ingot at processing temperatures in the range of 1470-1480°C at 1 and 2 minutes holding. This

is despite the lack of a 'conventional' thixoformable microstructure (i.e. spheroidal solid grains in a liquid matrix). Here, shearing may cause liquid pools to link up resulting in a slurry flow load as low as 1kN. Furthermore, despite thixoforming at very high liquid percent (about 50 to 80% according to DTA), heat loss (through radiation and convection) has produced a thin solid skin on the surface of the slug, preventing it from collapsing prior to processing.

The thixoforming load-displacement signals obtained indicate that, to achieve complete die filling, it would be desirable to have a low structure breakdown load in zone (2), and an almost constant or very slow rise of thixotropic flow load of zone (3). The work so far shows that these load signals are greatly influenced by processing temperatures and holding times. Further work on the optimisation of the thixoforming process and microstructural analysis in relation to the mechanical properties of the obtained parts is ongoing.

Acknowledgements

The authors wish to thank Dr Ian Todd for helpful discussions, Corus Engineering Steels, Rotherham, United Kingdom, for supplying the materials, and the National University of Malaysia (UKM), Bangi, Malaysia, for partly providing the financial support.

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Table.

Table 1

HP9/4/30	Chemical composition (wt.%)

	C	Ni	Co	Mo	Cr	Mn	Si	V	Cu	P	S	W
Standard	0.29-0.34	7.00-8.00	4.25-4.75	0.90-1.10	0.90-1.10	0.10-0.35	0.20	0.06-0.12	0.35	0.01	0.01	-
Chemical analysis	0.33	7.35	4.60	1.02	0.93	0.23	0.15	0.07	0.03	0.003	0.001	0.02

Figures.

Fig. 1

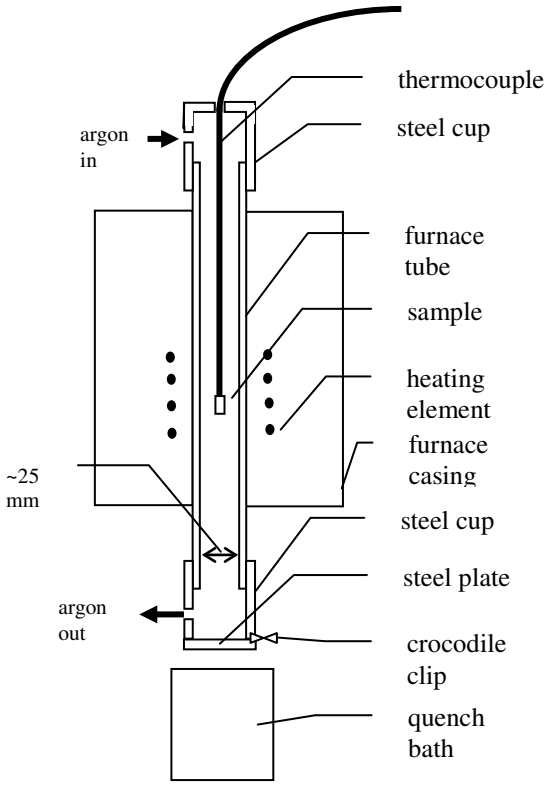


Fig. 2

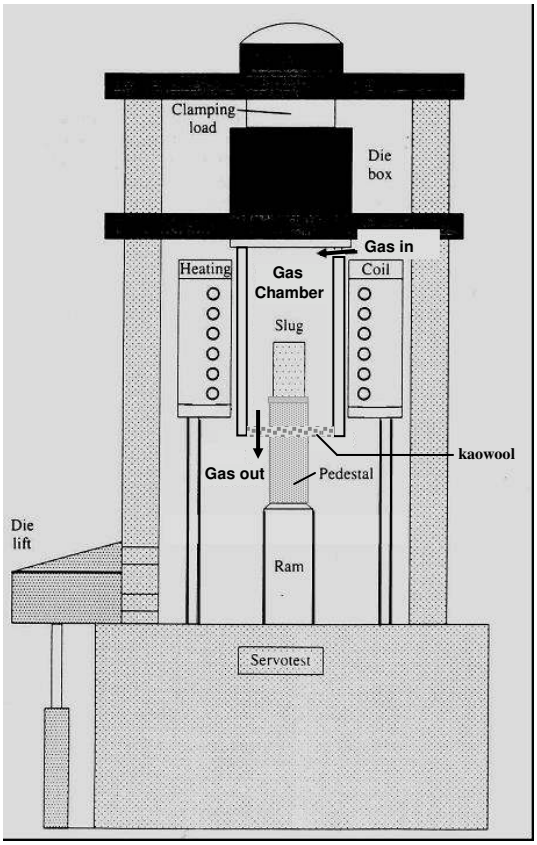

Clamping load
Die box
Gas in
Heating
Gas Chamber
Coil
Slug
kaowool
Gas out
Pedestal
Die lift
Ram
Servotest

Fig. 3

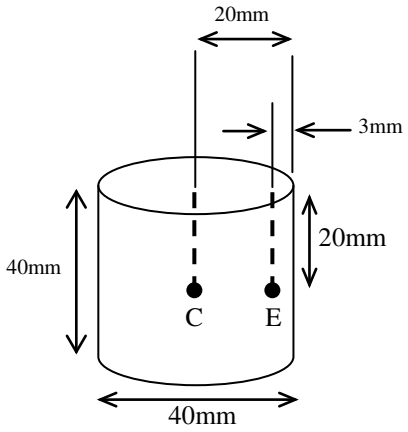
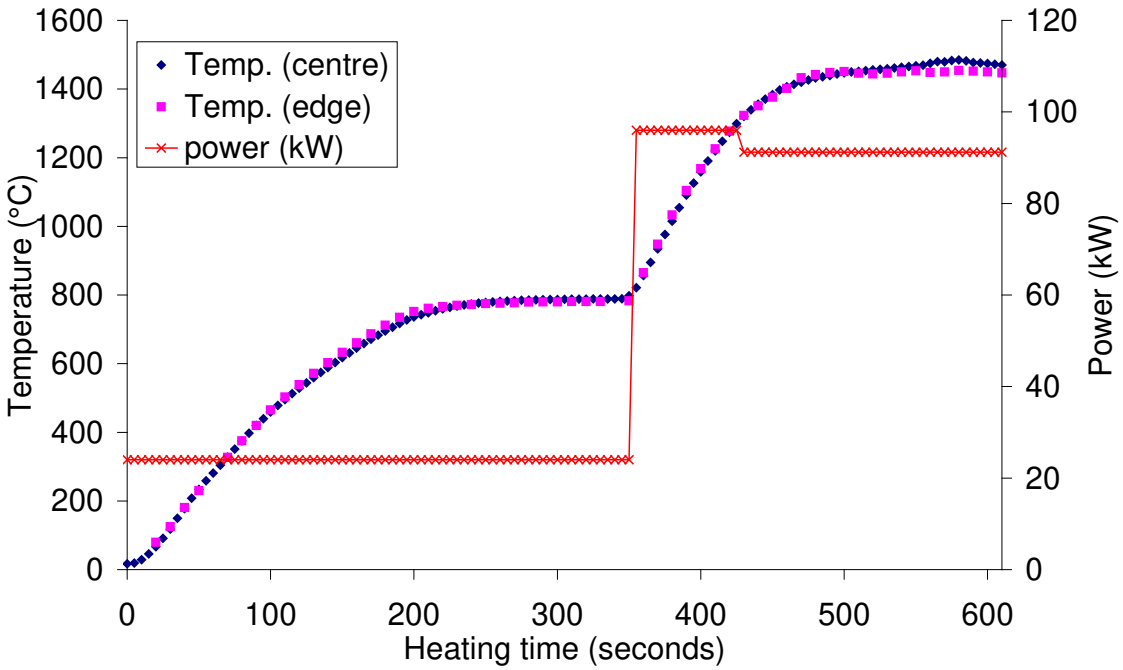
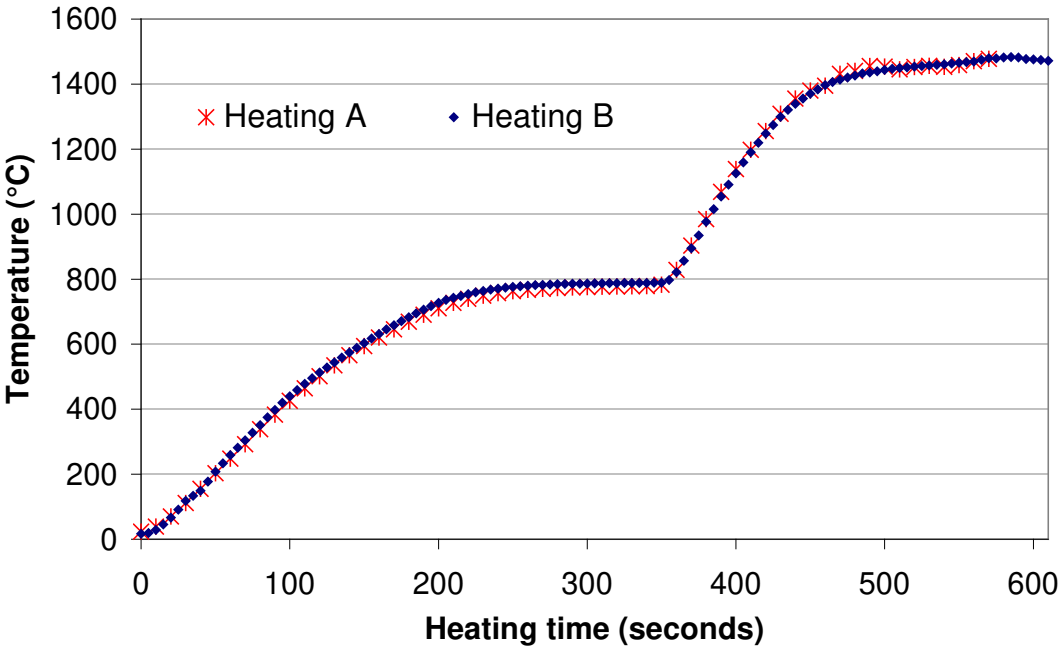


Fig. 4


Temp. (centre)
Temp. (edge)
power (kW)
Temperature (°C)
Power (kW)
Heating time (seconds)
0
200
400
600
800
1000
1200
1400
1600
0
20
40
60
80
100
120
0
100
200
300
400
500
600

(a)


Heating A
Heating B
Temperature (°C)
Heating time (seconds)
0
200
400
600
800
1000
1200
1400
1600
0
100
200
300
400
500
600

(b)

Fig. 5

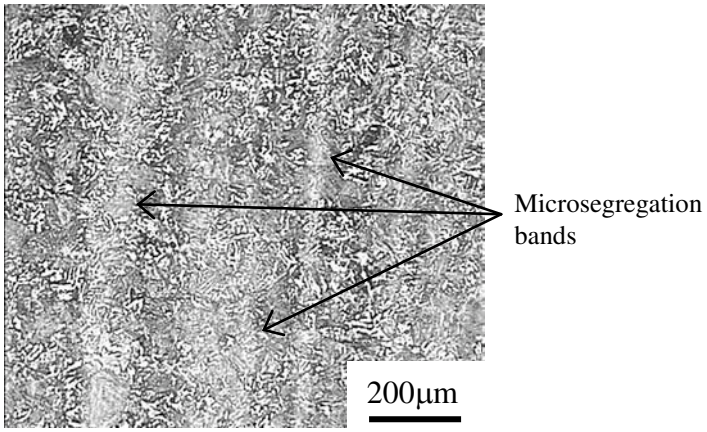


Fig. 6

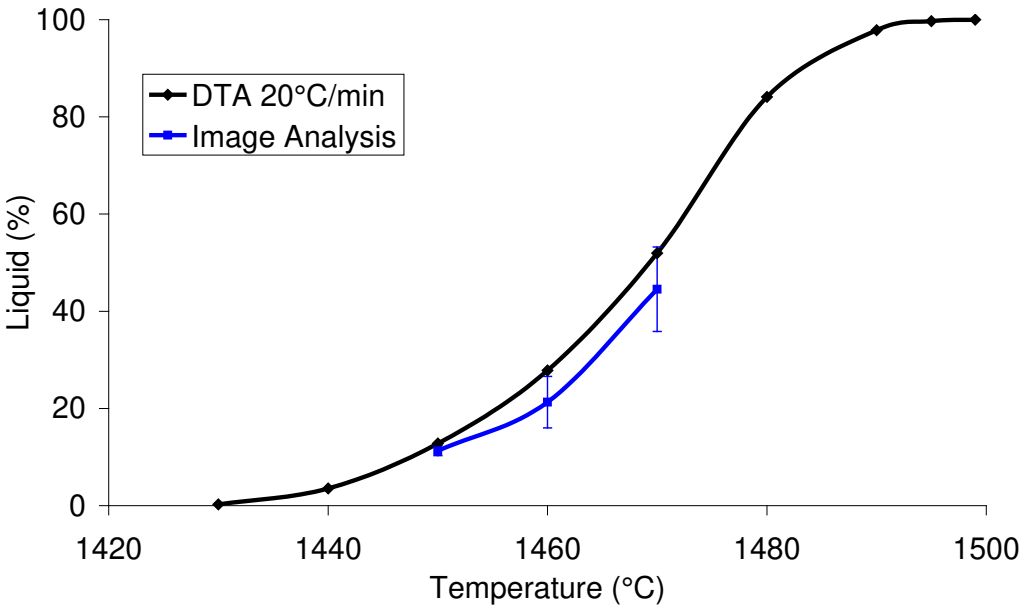

100
80
60
40
20
0
Liquid (%)
DTA 20°C/min
Image Analysis
1420
1440
1460
1480
1500
Temperature (°C)

Fig. 7

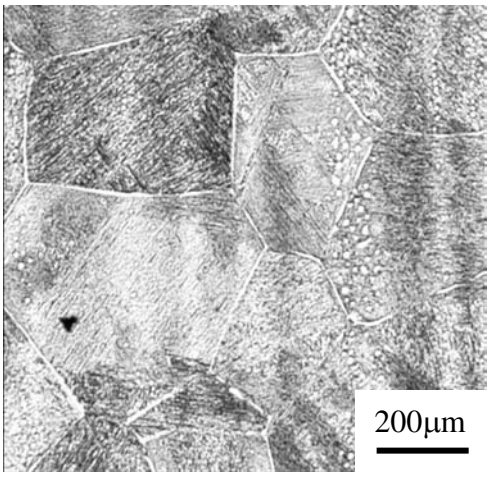
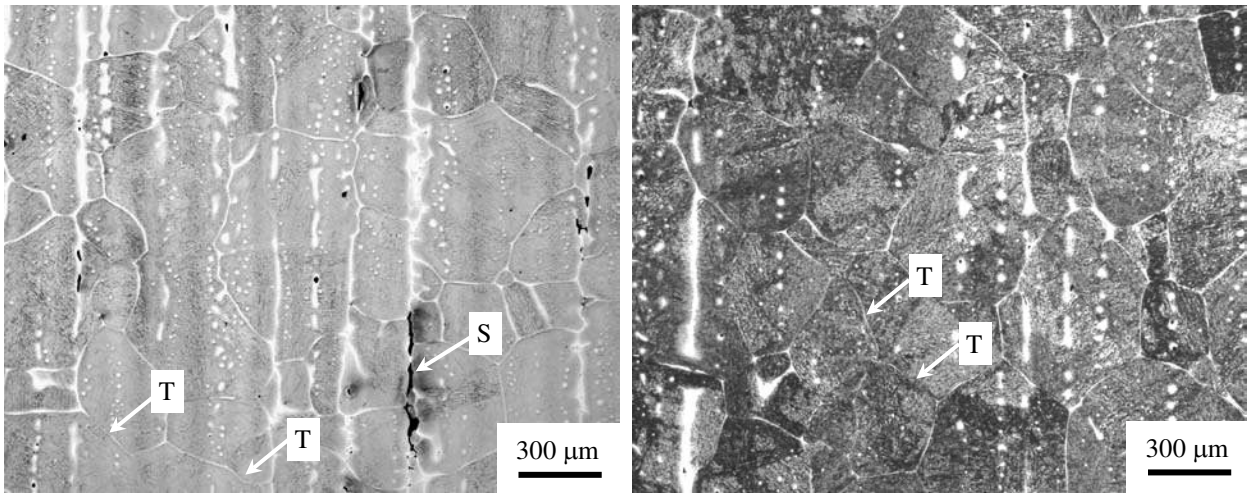
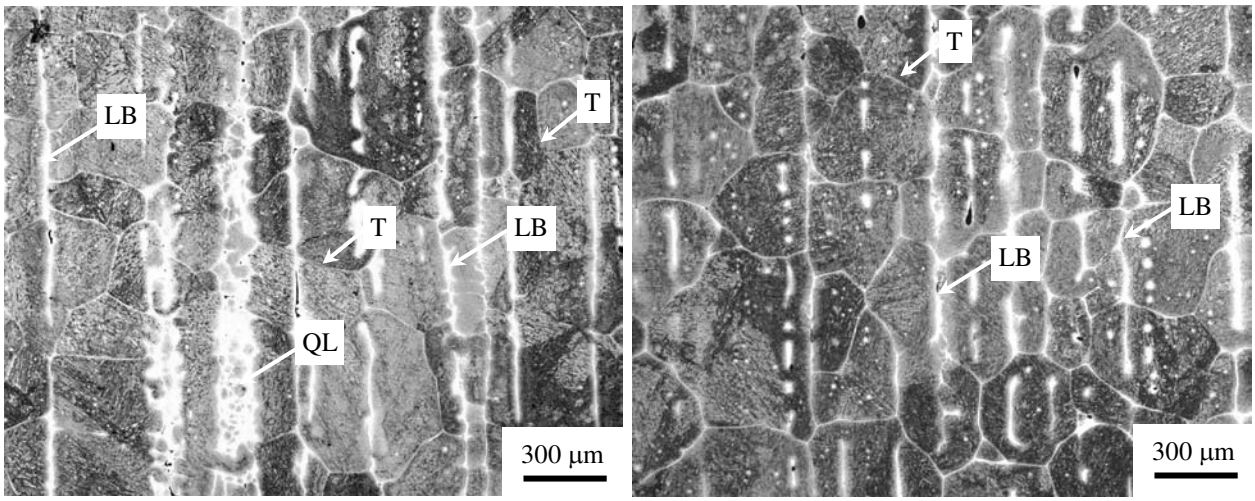


Fig. 8



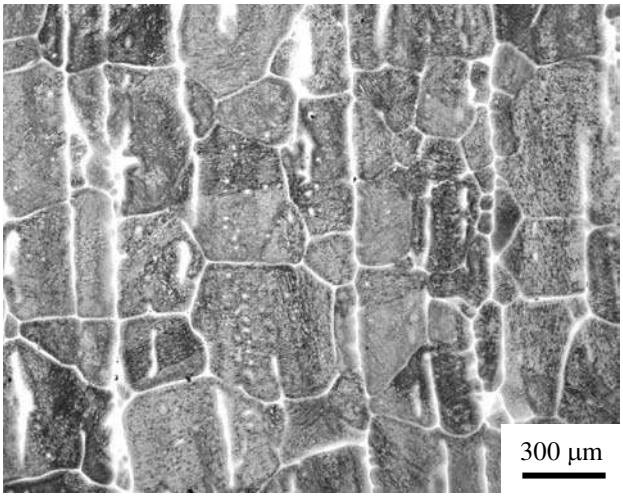
(a) (b)

Fig. 9

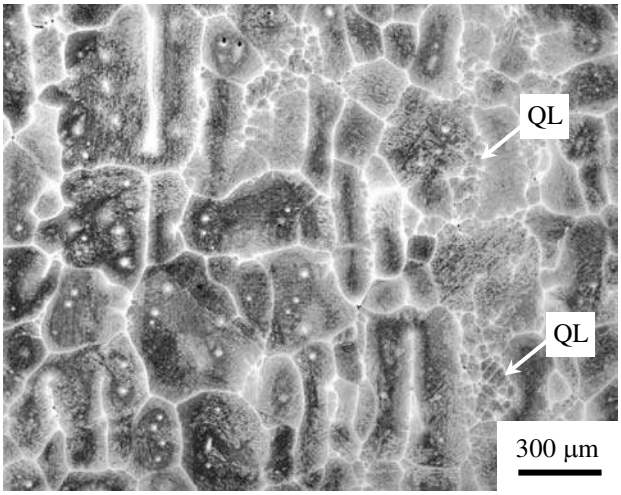


(a) (b)

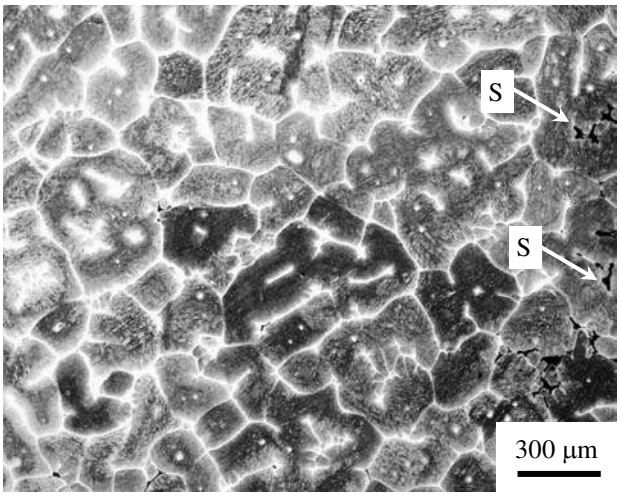
Fig. 10



(a) 2 minutes (longitudinal)



(b) 8 minutes, longitudinal

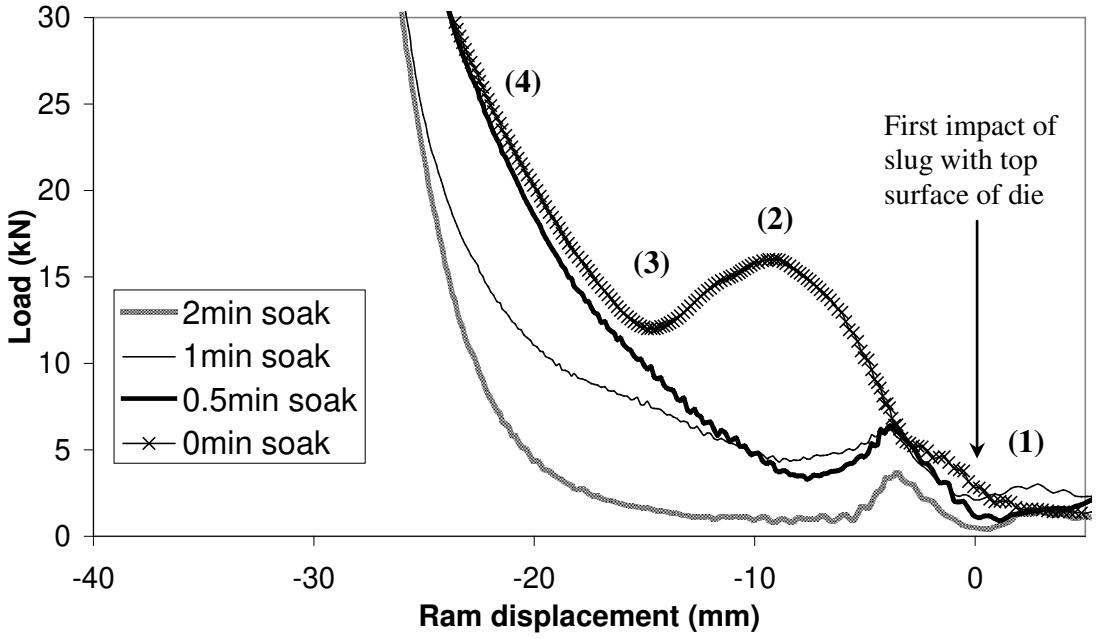


(c) 8 minutes, transverse

Fig. 11


cm 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30

Fig. 12


30
25
20
15
10
5
0
Load (kN)
-40
-30
-20
-10
0
Ram displacement (mm)
2min soak
1min soak
0.5min soak
0min soak
(4)
(3)
(2)
(1)
First impact of slug with top surface of die

List of Table and Figures.