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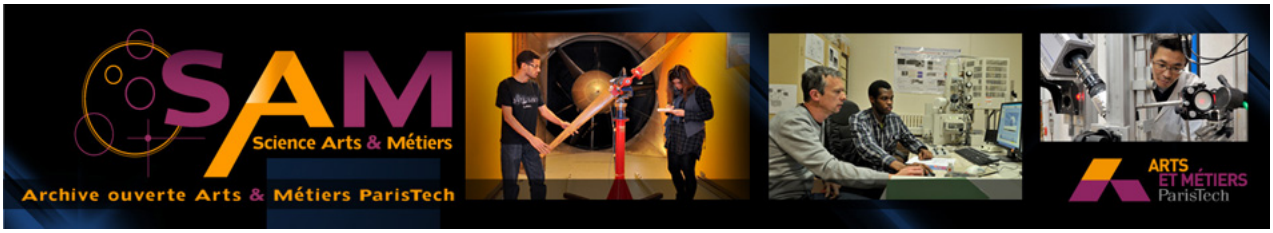

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Microstructure Evolution and Material Flow of Steel in Semi-solid Forming Process

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Abstract
The present study aims to identify and characterize the development of microstructure and deformation characteristics of steel grades in semi-solid state which is affected by the change in morphologies of microstructure at high temperature. Thixoextrusion tests with different combinations of forming temperature and forming speed were performed. It was identified that several process parameters, such as initial billet and die temperatures or forming speed, affect thermal exchanges thereby influencing the microstructure evolution and material flow. Furthermore, 2D and 3D microstructure characterization was performed on the same sample which was partial remelted and quenched. Reconstructed 3D images were compared with the ones obtained with a Scanning Electron Microscope and an Energy Dispersive Spectrometry system. The good agreement between 2D SEM observations and 3D X-ray microtomography results makes these two techniques efficient to characterize steels in the semi-solid state.

Keywords: Semi-solid forming, Microstructure evolution, Steel grade

Introduction

Semi-solid forming or thixoforming is an innovative technology in which the metallic alloys are processed in the semi-solid state to produce complex parts with high mechanical properties, usually in a one-step process with combined advantages of casting and forging. The semi-solid forming process on steels poses more technical problems, due to high working temperature and lack of understanding of the thermomechanical behaviour of materials in the given conditions, such as the location, volume fraction, size and shape of the liquid zones.

A great many parts have been thixoformed with various steel alloys and the process parameters, defects and mechanical properties of the parts were studied. Suggestions for process optimization and avoidance of defects were presented. Furthermore, the narrow process windows for billet preparation, reheating and forming cause a challenge for microstructure controlling. For this reason, details of the material behaviour, microstructure development and the physical basics of each process step need to be further studied and better understood. The aim of the present study is to provide the basic metallurgical and deformation characteristics of several steel grades in semi-solid state which is affected by the change in morphologies of microstructure at high temperature.

State of the Art

In thixoforging, a partial remelted billet with a predetermined liquid fraction adapted for the thixoforming process is sent into die tools. In order to decrease the thermal losses, the die tools were heated. During thixoforming, as stated by Favier et al. (2004), it is necessary to obtain in all points of the part a high speed deformation in order to ensure a homogeneous material flow at the semi-solid state, which could be achieved with a hydraulic press.

Spencer et al. (1972) first discovered the thixotropic behaviour in semi-solid metallic alloys when investigating hot tearing with a Couette viscometer. Thixoforming process for light alloys, such as aluminium alloys and magnesium alloy, has been industrialized. In the other hand, although the thixoforming of steel has become the focus of various research activities since the 1990s, it is still not introduced in industrial applications.

Puttgen and Bleck (2004) performed a great many DTA experiments on various steel grades, identifying that the X210CrW12 and HS 6-5-3 tool steels are suitable for the process for the reason that they have a large freezing range. The bearing steel 100Cr6 is also suitable.

Various routes for billet preparation have been applied to obtain the primary billets with non-dendritic microstructures. Meanwhile, the adapted steel grades have been manufactured for thixoforming. For example, C38LTT (Low Thixoforming Temperature) produced by ASCOMETAL has a reduced solidus temperature.

Becker (2008) illustrated the possibility of performing thixoforging experiments on a rolled C38 bar. Li et al. (2008) identified that the inverse peritectic reaction can lead to spheroidisation in stainless steels in the semi-solid state. Finally, Omar et al. (2009) discussed the potential route for thixoforming microstructures by directly reheating from the as-received condition into the semi-solid zone.

Experimental Procedure

Two types of steel grades were investigated in this study: C38 low carbon steel grade and M2 high speed tool steel. Billets of C38 were thixoextruded to study the influence of process conditions on the material flow. Becker (2008) mentioned that the process conditions have a great influence on the thermal exchange between billet and die, which in turn has an effect on the liquid ratio, on the microstructure and therefore on the material behaviour. Liquid fraction is one of the most important parameters. As it is difficult to identify the former liquid zones in the quenched C38 samples, billets of M2 were partially remelted and quenched for the purpose of studying the

microstructure evolution during reheating step. Billets were cut from rolled bars of 30mm and 38mm in diameter.

Fig. 1 shows the liquid fraction curves for C38 (Rouf, 2003) and M2 (Puttgen, 2007) as a function of temperature; they were determined by Differential Scanning Calorimetry (DSC) and Differential Thermal Analysis (DTA), respectively. Here, the values given are approximate, because in the DSC/DTA tests, the specimen size and heating rate are quite different from the ones used in thixoforming process. In thixoextrusion experiments, average heating rate is about 17 times bigger than the one used in DSC and DTA tests.

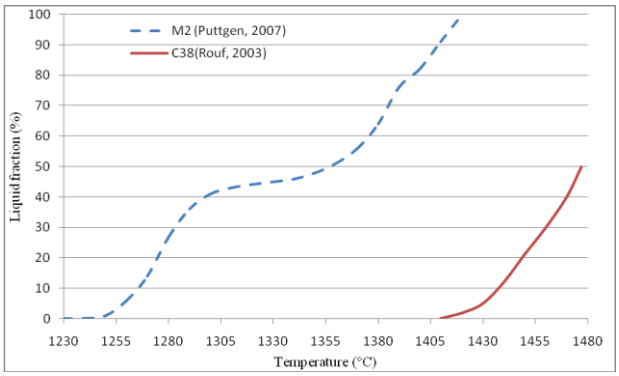


Figure 1. Liquid fraction curves for C38 and M2 steel grades as a function of temperature by means of DSC and DTA measurements at a heating speed of 20 K/min.

The chemical compositions of these two grades are given in **Table 1**.

Table 1. Chemical composition of the C38 low carbon steel and M2 high speed tool steel (weight 10^{-3} %, iron balance)

	C	Mn	P	S	Si	Al	N	Ni	Cr	Cu	V	W
C38	418	751	10	21	198	21	65	77	144	133		
M2	850	250			35				410		190	64

Due to its better repeatability and the lesser billet heating time, induction heating technique was used in our experiments. In the aim of studying the material flow and the microstructure evolution, thixoextrusion experiments were carried out on low carbon steel C38 in an extrusion device as shown in **Fig. 2** (Becker, 2010). As illustrated in the figure, the induction coil is located above the die gate in order to reduce the heat losses during the transfer of the billet and to retain the billet shape. A shock absorption system was developed and integrated into the tools to avoid further material deformation during the slowdown in punch speed.

Before performing the thixoextrusion tests, the billets of C38 were isothermally induction heated in a protective argon atmosphere using a multistage heating cycle. Before forming, the billet temperature was measured by four thermocouples which located in different places.

Billets of C38 were thixoextruded from 30mm to 12mm in diameter with various process conditions which are given in **Table 2.**

Table 2. Experimental conditions

Conditions	Values	
Billet temperature (℃)	1429 - 1451	
Punch speed (mm/s)	30	215
Die temperature (℃)	Room temperature	400

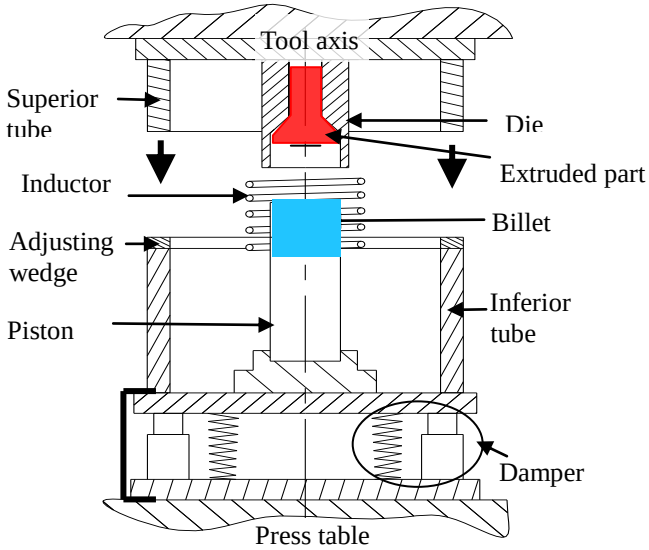


Figure 2. Complete extrusion device designed in Arts et Métiers ParisTech Metz.

In addition, another steel grade was tested. A billet of M2 steel was just non-isothermally induction heated for the purpose of studying the microstructure evolution during reheating step and subsequently water quenched. During heating, the billet was partially inserted in the induction coil with a part sitting on a ceramic holder, to ensure the coexistence of solid and semi-solid states.

The HCL and *Bechet-Beaujard* etchants were used to observe the macrostructure of thixoextruded parts, while Nital 3% was used for microscopic characterization. The microstructure investigations were carried out on both grades using optical microscope and a Jeol 7001FLV Scanning Electron Microscope (SEM). Energy Dispersive Spectroscopy (EDS) analyses were performed with an Oxford INCA System to study the elemental distribution of M2 steel.

Results

C38 low carbon steel. Billets, directly heated to semi-solid state from as-received state (hot rolled) were thixoextruded with various process conditions. The punch speed, billet and die temperatures influence the process results, such as forming load and part shape (Becker, 2010). The microstructure evolution and material flow will be presented as follows.

Material in the initial state. An optical micrograph of the as-received low carbon steel C38 is shown in **Fig. 3.** The material has been rolled at high temperature, leading to a homogeneous fine microstructure composed of ferrite and pearlite that can be observed in **Fig. 3.**

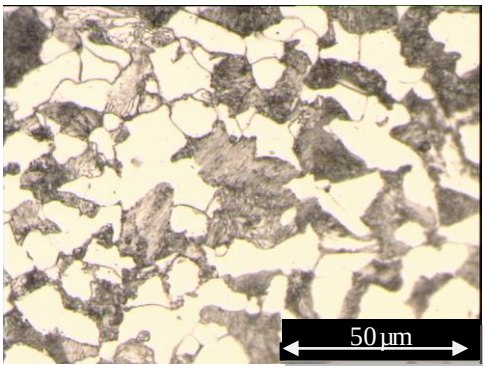


Figure 3. Microstructure of as-received C38 (Becker, Arts et Métiers ParisTech).

Thixoextrusion. **Fig. 4** shows the macrostructures of two thixoextruded parts, considering the process conditions of 40CM (40: punch speed = 40 mm/s, C (cold die): die temperature = 30 ℃, M (median): initial billet temperature = 1437 ℃) and 200WH (200: punch speed = 215mm/s, W (warm die): die temperature = 400 ℃, H (high): initial billet temperature = 1445 ℃), respectively. The parts were macroetched with HCL.

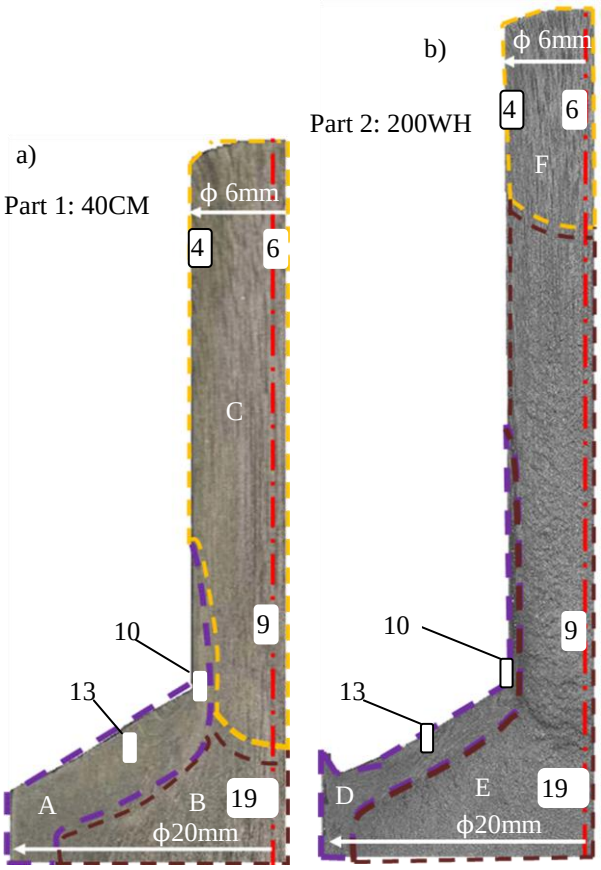
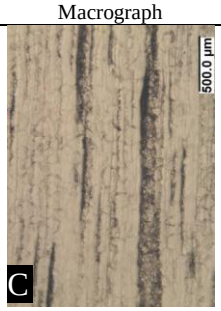
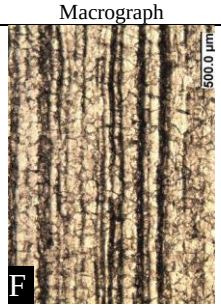
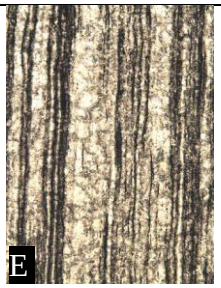
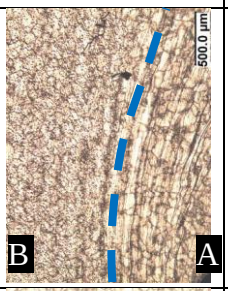
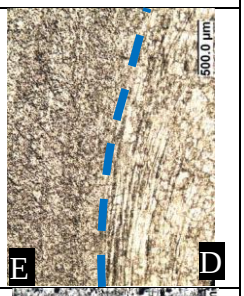
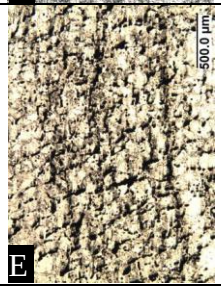


Figure 4. Macrostructures combined with simulation results thixoextruded with various process conditions in (a) Process condition is 40CM and in (b) Process condition is 200WH (Becker, 2008).

Three different zones, being separated by different flow behaviour, were observed in each thixoextruded part. These zones were marked with A, B, C and D, E, F for Part 1 and Part 2, respectively. By comparing the zones with the same material flow in the two parts, the thickness of zone A is less than zone D, zone C is longer than zone F in longitudinal direction and the volume of material in zone B is smaller than the in zone E. Besides, the length of part 1 is smaller than part 2. In order to figure out the mechanism responsible for the material flows, macro and microexaminations were performed on different zones.

Representative macrographs from various zones in the parts were shown in **Table 3.**

Table 3. Macrographs and micrographs of different points in parts 1 and 2

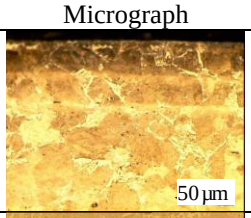
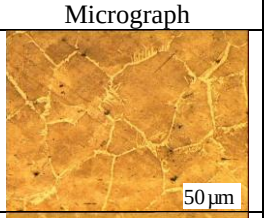
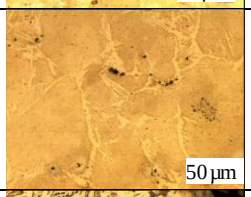
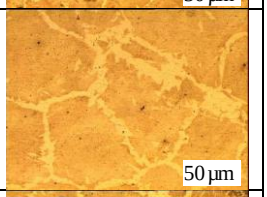
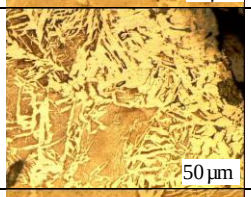
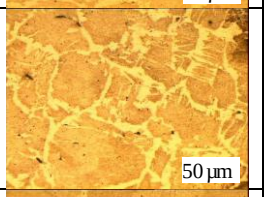
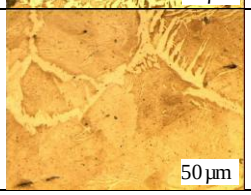
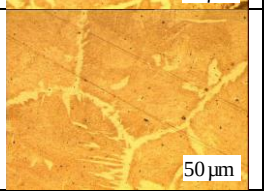
Observation point	Part 1: 40CM	Part 2: 200WH
	Macrograph	Macrograph
6		
9		
10		
13		
19		

Material flow is illustrated by the macrographs from various zones in the parts. The thixoextruded parts were Bechet-Beaujard etched.

During extrusion, the material in zone A and zone D remains static and in contact with the tool walls. Formed structure in zone A can be seen in the macrograph of 13. It is caused by the high level temperature exchange between the cold die and the billet. During forming of the billet at a low speed and with a low die temperature, heat exchange reduces the billet temperature which affects the liquid fraction and therefore the viscosity of material. An extrusion flow direction was observed in zone C or F illustrated by the macrographs of 6 and 9 in **Fig. 4 (a),** or 9 in **Fig. 4 (b).** This confirms the classical material flow induced by extrusion. As shown in macrograph of 19, the material in zone B or E maintains the semi-solid properties. The material in zone B and E does not exhibit the "fiber" flow; there are waves in the zones which correspond to the semi-solid material with a fluid behavior.

Grain size and microstructure were studied from the micrographs obtained in different points, the structure of which is summarized in **Table 4.**

Table 4. Micrographs of different points

	Part. 1	Part. 2
Position	Micrograph	Micrograph
4		
9		
13		
19		

The grain size depends on the temperature. During thixoextrusion, the forming speed and die tool affect the material temperature and therefore influence the grain size. Moreover, the nucleation of large grains is exhibited in hot zones or the zones with less heat exchange.

M2 high speed tool steel. In this part, the microstructure was studied on as-received part and the part quenched from semi-solid state.

Material in the initial state. As shown in **Fig. 5** with SEM micrographs of the as-received M2 steel (cross sectional and longitudinal surfaces), fine carbide particles are distributed at grain boundaries and in the matrix; the average grain size is 7-12 µm. Microsegregation bands caused by the rolling process are also observed on longitudinal surface. Two different types of carbides are present. EDS analyses show that the whiter particles are M_6C-type carbides which are rich in tungsten and molybdenum, while the darker ones are vanadium-tungsten-molybdenum rich MC-type ones.

Microstructural development during partial remelting. The billet was induction heated. A pyrometer was used to measure the local temperature. When the sample reached the redefined quenching temperature, it was subsequently quenched to freeze the microstructure presented in the semi-solid state. For the purpose of studying the microstructrual evolution, the billet position in the induction coil is shown in **Fig. 6.** The setting up is realized for the coexistence of the material in both semi-solid and solid states.

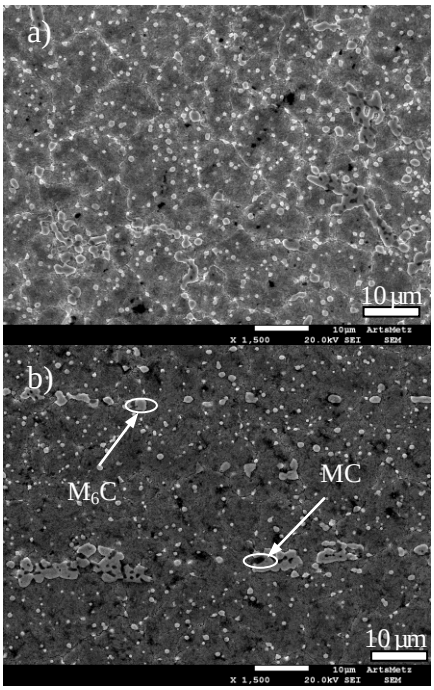


Figure 5. Microstructures on (a) cross sectional surface and (b) longitudinal surface.

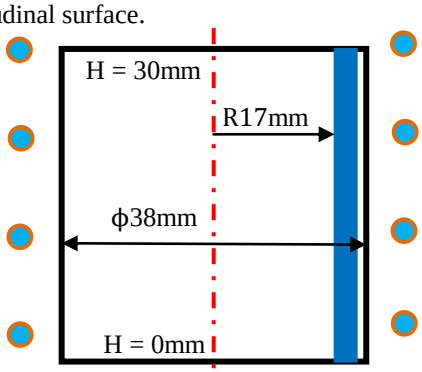


Figure 6. The position of billet during induction heating (experimential procedure)

The microstructure changes depend on the height. At the bottom of the billet, it remained in solid state during induction heating. The obtained microstructure is shown in **Fig. 7 (b)**; it is similar to the one observed in the as-received material. With increasing height, the temperature increases. At the height of 5mm (**Fig. 7 (c)**), the majority of the carbide particles are still distributed in matrix and bands. Only the grain size is bigger which results from the elevated temperature. However, as the temperature rises, most of the original carbide particles have been dissolved and new ones are precipitated at the grain boundaries, as shown in **Fig. 7 (d)**. With increasing temperature, the carbides along the microsegregation bands starts melting, as shown in **Fig. 7 (e)**. At the same time, the grain keeps growing. However, the growth speed in the direction parallel to the working direction is bigger than in axial direction, which is related to carbide pinning.

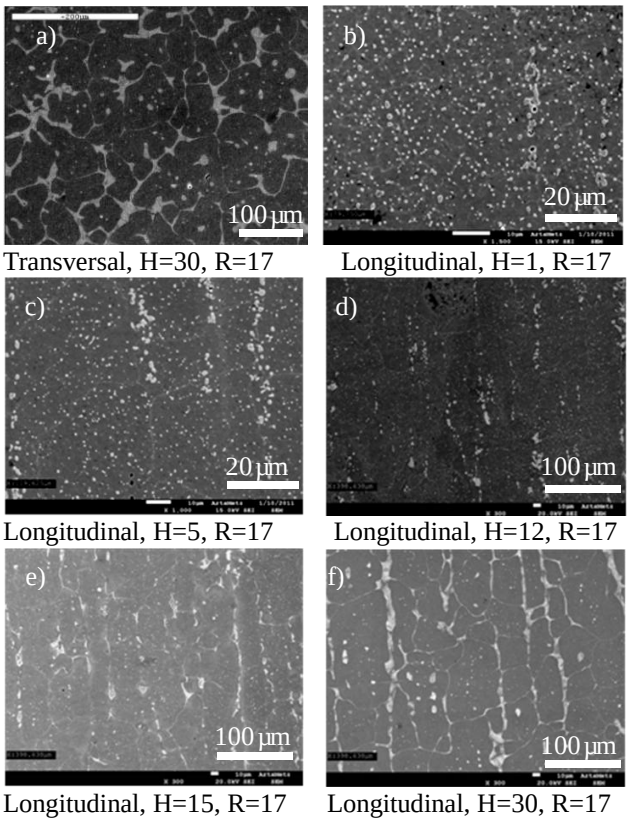


Figure 7. SEM micrographs of as-received M2 steel induction heated to semi-solid state

3D microstructure characterisation. High energy X-ray microtomography is a non destructive technique which allows determining the total liquid volume fraction, the entrapped liquid fraction and the liquid volume distribution. The first trial of this technique to characterize the microstructure of semi-solid steel grade was performed on M2 steel grade. Experiments were performed in European Synchrotron Radiation Facility ESRF, ID15 beamline. The final spatial resolution is about 1.5 µm. The heated specimens in the semi-solid state were quenched in order to "freeze" the microstructure at room temperature, preventing alloying elements to diffuse in the steel from liquid zones.

After the tomography, the image processing software ImageJ was used to reconstruct the different projections. 3D images of as-received and partial remelted M2 steel are presented in **Fig. 8.**

Carbides are presented as white zone in the images. In **Fig. 8 (a)**, the carbide particles are still isolated distributed in bands, while the connected carbide could be observed in **Fig. 8 (b).**

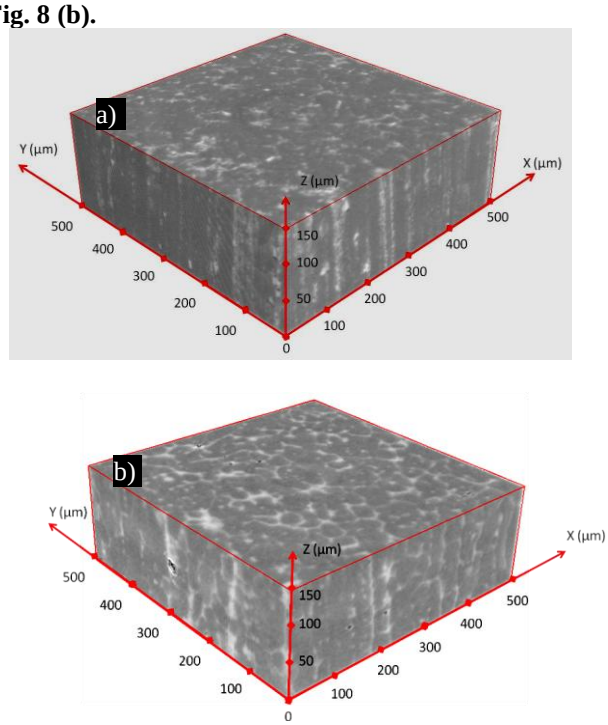


Figure 8. 3D representation of M2 steel in (a) as-received state, and in (b) partial remelted state.
Volume = 0.5mm×0.5 mm×0.15 mm

Discussion

Thixoextrusion experiments on C38 steel indicate that the process parameters such as punch speed, initial billet and die temperatures affect the material flows and the development of microstructure during forming operation.
The material flows are controlled by liquid fraction, distribution of liquid, microstructure... These factors are also affected by the billet temperature, die temperature, heat exchange and other process parameters. The influence of any changes of process parameter will eventually act on the fraction and distribution of liquid, and therefore on the microstructure and the mechanical properties. In this study, three types of flow were shown: static flow in zone A or zone D, semi-solid flow in zone B or zone E, typical forging flow in zone C or zone F. The length or thickness of these zones is largely dependent on the temperature, the heat exchange and punch speed. At the same punch speed, the increasing of the die or billet temperature will reduce the thermal exchange, therefore increasing the size of semi-solid zone. In the same way, increasing the punch speed decreases the thermal exchange time which relevantly reduces the loss of heat. However, increasing temperature leads to increasing liquid fraction which results in the

reduced forming load, and to grain growth, that causes the microstructual coarsening (Atkinson, 2008) which in turn reduces the mechanical properties of parts.

When studying the micrographs of thixoextruded parts, it was found that the grain size in semi-solid zone is bigger, which is due to the increased temperature.

In the micrographic study of M2 steel, the partially remelted material exhibits appearance of a microstructure which is different from the as-received material. The grains are bigger, and the solid phase is surrounded within a liquid matrix. During heating, major liquid appearance occurs in the microsegregation bands; these bands can form interconnected networks with each other and with entrapped liquid, as shown in **Fig. 7 (a) and (f).** This could be explained by two coarsening phenomena: Ostwald ripening and coalescence. The reason why the microstructure in semi-solid state does not exhibit a conventional globular structure could be due to less holding time. Still, the structure is suitable for thixoforming process with thick liquid films formed at grain boundaries.

In addition, X-ray tomography has been proved to be a powerful tool to provide data that are representative of the semi-solid state; no such experiments have been carried out on semi-solid steel grade before. In the microtomography study on M2 steel, the specimens were observed at room temperature after quenching, with a relatively high resolution of about 1.5 µm.

The tomography results are in good agreement with SEM micrographs, with corresponding grain size and liquid spatial distribution. Some differences can be observed in as-received state; the size of many carbide particles is smaller than 1 µm, which makes them impossible to distinguish with the resolution of X-ray tomography. In contrast, when the distance between two carbide particles is less than 1.5 µm, the isolated carbide particles will be considered as one.

Conclusion

Thixoextrusion experiments were carried out with various process conditions. The macrostructure and microstructures of thixoextruded parts were studied. It was identified that several process parameters, such as initial billet and die temperatures or forming speed, affect thermal exchanges thereby influencing the microstructure evolution and material flow.

The microstructures of M2 high speed tool steel in the as-received condition and in the semi-solid state have been studied in 2D and 3D. The results obtained using different techniques were successfully compared. The good agreement between 2D SEM microstructures and 3D X-ray tomography results makes these two techniques efficient to characterize steels in the semi-solid state.

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