ESRF	Experiment title: Liquid and solid repartition in steels during thixoforging process using X-ray microtomography	**Experiment number**: MA-975
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Report:

Aims of this study

Thixoforming process combines the advantages of forging and casting process for obtaining parts with good qualities. It has been industrialised on low melting point materials, such as aluminium and copper. However, for thixoforming of high-temperature alloys such as steel, the high working temperature and lacks of understanding of the thermomechanical behaviour cause difficult problems to solve. The total liquid volume fraction, entrapped liquid fraction and liquid distribution are important parameters that remain to be quantified, because the cohesion of the solid skeleton can affect the thixoforming process. By means of 2D observations, the fully characterization of microstructures is not sufficient. It is thus crucial to carry out X-ray microtomography experiments to quantify in 3D the liquid volume fraction and distribution with sufficient accuracy during the thixoforming process, for a better understanding of this process on steel grades.

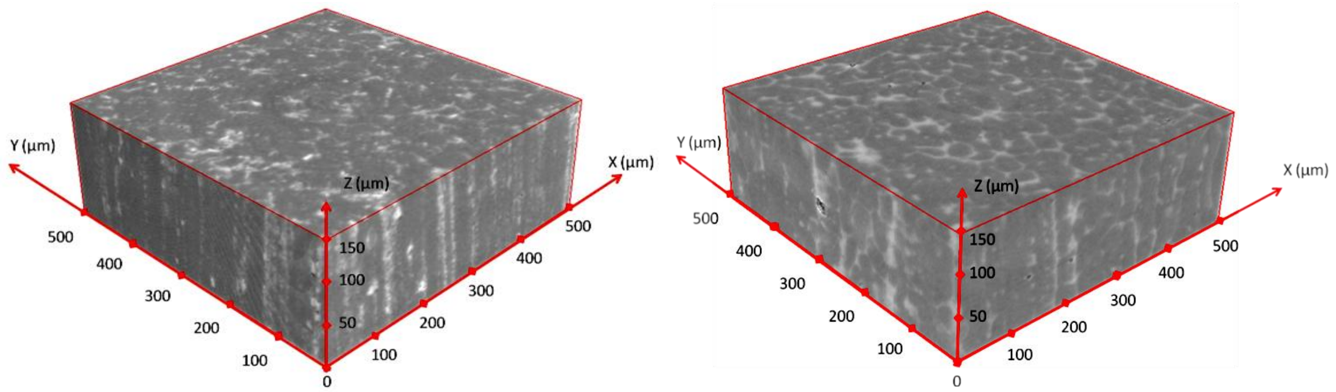
Experimental procedure

X-ray microtomography experiments were carried out on several steel grades (M2, 100Cr6 and 316L) with different thermal histories to characterize the microstructures with a resolution of around 1.5µm. These experiments were performed using samples of M2 and 100Cr6 water-quenched from semi-solid state, thixoforged samples of grade 100Cr6, and as received samples sectioned from rolled bars (M2, 100Cr6) and wires (316L). All the samples are cylinders 1.2mm in diameter. The energy used was around 60keV, with a spatial resolution of about 3µm. The number of projections applied in our tomography experiments was 3600. Furthermore, in situ microtomography experiments were finally performed on M2 steel directly at high temperature (1400°C), as a trial.

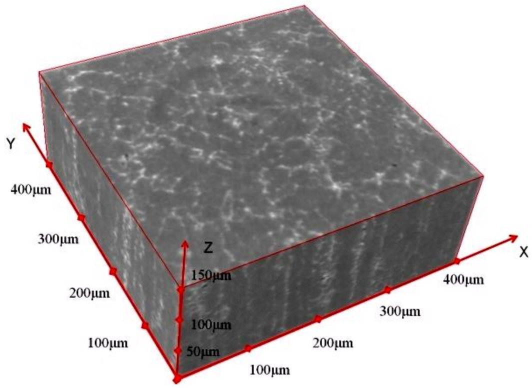
The results and discussion

With the software ImageJ, it's possible to visualize the reconstructed slices obtained by X-ray microtomography. Fig. 1 shows typical 3D reconstructed volumes of M2 steel in different states. When looking at these three images, the white zones are richer in alloying elements (V, Mo and W) which lead to a larger X-ray absorption. The distribution of these zones (location and size) is different in the initial state, at high temperature and after quenching, although white segregated bands can be observed in all images. The connected carbide networks are observed in Fig. 1 (2) and (3). Furthermore, some cavities can also be identified from the images.

In fact, since the liquid zone is richer in alloying elements than the solid part of the material, it is possible to detect such zones during in situ analyses and ex situ analyses on quenched samples. In the latter case, the alloying elements have no time to diffuse in the material because the cooling is very fast. Tomography allows then to get several parameters which are not easy to characterize in 2D, such as the total volume fraction of liquid, its volume distribution, the fraction of entrapped liquid and its location, the volume fraction of cavity/porosity… For the particular microstructure of Fig.1 (2) and (3), the volume fraction of liquid are 21.07% and 13.32%, respectively.



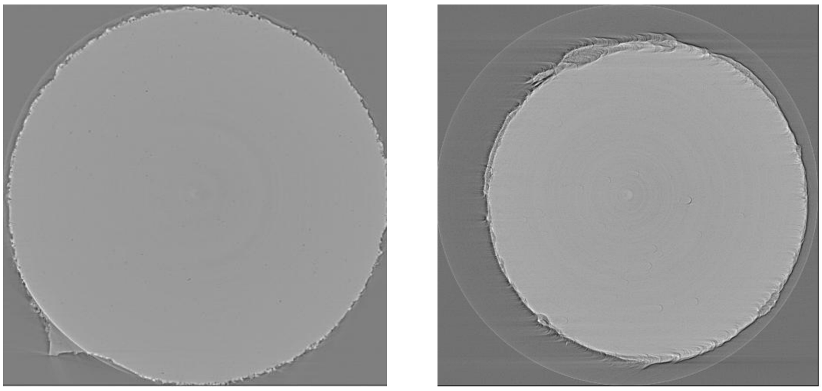
(1) M2 tool steel in initial state (2) M2 tool steel quenched from semi-solid state



(3) M2 steel in the semi-solid state (in situ tomography at high temperature)

Fig 1. 3D representations of M2 steel

However, when performing the X-ray tomography on 100Cr6 and 316L steel grades, it is unfortunately impossible to observe different phases, Fig. 2. The quantity of alloying elements is too small or their atomic number is too close to have a sufficient absorption contrast between liquid and solid parts.



(1) 100Cr6 (2) 316L

Fig 2. X-ray microtomography images of 100Cr6 and 316L steel grades

Conclusion and future proposal

X-ray microtomography has been proved to be a powerful tool to well characterize the semi-solid state of M2 steel. The former liquid zones in M2 steel could be well detected during in situ testing and in quenched samples. Since the in situ analyses were just performed as a trial during the last allocated shift - successful analyses with a homedeveloped device -, it would be very interesting to submit another proposal (ID15 beamline) in order to make more similar experiments. It is the only technique which enables to quantify in 3D the volume fraction of liquid at different high temperatures and to better understand the microstructural changes during holding in the semi-solid state and thixoforming process.