Report on the experiments performed at ESRF Beamtime ME 1188

Subject : "In situ characterization of bainitic transformation under stress by high energy X-ray diffraction".

Participants :
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The aims of the experiments were to realize an in situ study of the phase transformation under stress using high energy X Ray diffraction, and to further analyse the role of the applied stress on the behaviour of the alloy during the transformation.

In this way, a defined thermomechanical cycle needed to be applied to the specimen while structural information were collected simultaneously by X-ray diffraction. In situ characterization of the transformation kinetics by high energy X-ray diffraction was previously conducted considering only thermal evolutions[1].

To perform these experiments a tensile machine from FAME38 designed for applying thermal and mechanical variations was available. In our case, we needed to heat the specimens up to about 1050°C for a few minutes and to cool it to the transformation temperature. This heating was necessary to have the phase transformation on heating before the one on cooling.

The available tensile machine was the ETMT from FAME38.
The heating is performed by DC current, which intensity is controlled according to the programmed heating rate. The stress (or displacement of the grip) is applied via an electric actuator.
Concerning the actuator the maximum load is 5kN
Concerning the heating, the specifications of the device are
maximum tension 8V, maximum intensity 450A.
The tensile specimens were designed considering the specificities of the machine and the aim of our experiments. Due to the increase in grain size on heating, we needed to analyse specimens with a width of at least 4 mm. The section of the specimen was then defined as 4x2 mm^2.
With this defined geometry, preliminary tests were successfully realised in July 2005 by Guillaume Geandier. The heating power of the machine was large enough to bring the specimen in the austenite temperature range.

During the beam time, experiments were conducted in several conditions. Unfortunately the heating device did not allow heating the specimens to the high temperature range necessary for the achievement of the experiment. Indeed, a DC alimentation breakdown prevented the voltage to exceed 1.5V, and leaded to a dramatic switch off the heating power.

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[1] Austenitization and precipitate dissolution in high nitrogen steels : An in-situ high temperature synchrotron X-ray diffraction analysis using the Rietveld method.
A. BENETEAU, P. WEISBECKER, G. GEANDIER, E. AEBY-GAUTIER and B. APPOLAIRE.
Materials Science and Engineering A, Volume 393, Issues 1-2, 25 February 2005, Pages 63-70

In these conditions, it was impossible to reach temperatures higher than 600°C as shown on figure 1 in which we reported the temperature evolution with time of an attempt.

Various attempts have been done to repair this breakdown or to get round it with the help of the ID15 technical staff as with Matthew Peel. The apparatus being under guarantee nothing could be undertaken on the electronic part of the device and the attempts, consisting in reducing the voltage or decreasing the section of the sample, revealed fruitless.

From our tests different comments could be pointed out :

- The program and control of the thermal cycle and of the load are efficient.
- The DC alimentation breakdown of the ETMT device, limiting the voltage to 1.5V (instead of 8V as indicated in the technical specifications of the device) was the main problem, preventing our experiments to be carried out properly. The experiments could have been achieved conveniently without this limitation.
- Finally one has to mention too that the use of the DC current heating leads to some error in the measurement of the temperature. As the thermocouple is spot welded on the surface of the specimen, an additional tension associated with the DC current is measured and leads to an error. This error was estimated (depending on the way the welding is done it could reach 50°C at the higher temperature) and has of course to be considered in the experiments. To overcome this problems measurement of the temperature without spot welding the thermocouple should be achieved.

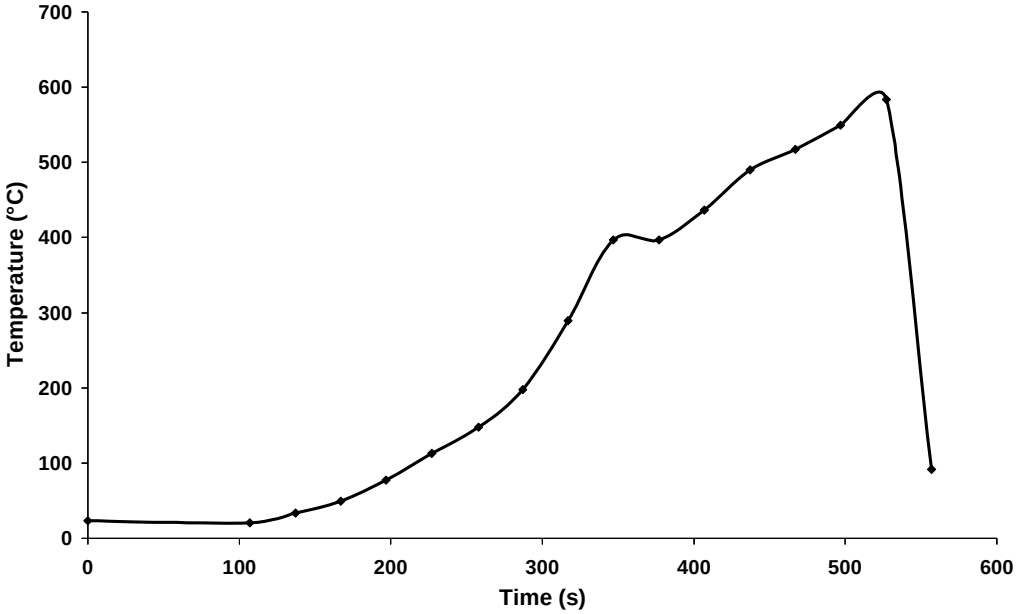

Figure 1 : Temperature evolution with time for the specimen heated with the thermomechanical tensile machine.