In-situ observation of solid-solid phase transition in metals at high temperature

Alexandre Fournier*[1,2,3], Michel Coret[2], and Nicolas Bruzy[1,3]

[1]CEA DAM – CEA DAM Bruyères le Châtel – France
[2]Institut de Recherche en Génie Civil et Mécanique – 1 Institute of Civil Engineering and Mechanics (GeM), Centrale Nantes, Nantes, France – France
[3]LMCE – Université Paris-Saclay, CEA, LMCE, F-91680 Bruyères-le-Chatel – France

Abstract

Plasticity and phase transitions are among the main deformation mechanisms of metals. While each of them is understood separately, the coupling between them is less obvious. This coupling is challenging both for modeling (a multiscale model, from the atomic to the mesoscopic scale is needed) and experiments (one has to combine a good time resolution, adapted to the phase transition kinetics, and a good spatial resolution at the microstructure scale).This work focuses on solid-solid phase transitions in temperature. We present a device that allows for their in situ observation.

The sample is placed in the middle of a vacuum chamber, to avoid oxydation and to prevent the heat haze phenomenon (1). An electric current heats the sample by Joule effect. This allows for a wide range of temperature rates from no heating to several hundreds of degrees per second. Strain fields at one surface of the sample central zone are obtained thanks to an optical camera and the digital image correlation technique. The other side of the sample is used to measure the temperature of the sample using two pyrometers, one for low temperature and one for high, both focused at the center of the sample. A PID controller regulates the power intensity so that various temperature profiles can be imposed. A limit of the experimental procedure is that the temperature field is not perfectly homogeneous although it is supposed to be. The main temperature gradient is in the length of the sample, due to the grips acting as heat sinks.

In order to perform digital image correlation, it is necessary to have strong grey level textures on the images. To achieve this, a speckle pattern is applied on the studied surface of the sample. Its creation is a challenging because it must neither be too sparse nor too dense. In our case, the deposition is done thanks to an airbrush spraying a mixture of alcool and fine alumina particules (approximately 1μm in diameter).

In order to characterize the microstructure of the samples, the EBSD (for Electron Backscatter Diffraction) technique is used. It allows the measurement of crystals orientations on the sample surface, therefore detecting the grain sizes, positions and boundaries in a polycristal. Before performing the EBSD, the sample is polished in order to obtain a surface as flat as possible. The next step consists in making small marks around the soon-to-be mapped

*Speaker

surface in order to be able to identify the area location later.

The sample is analysed by EBSD at ambient temperature before and after the transformation. Coupled with the digital image correlation data, an analysis of the sample in the high temperature phase can be made, and hence improving the understanding of phase transitions.

This procedure is illustrated on iron. The cristallographic structure of pure iron changes at 910∘C. Under Tt=910∘C, iron is in the so-called α phase, and has a body-centered cubic (or BCC) cristallographic structure. It is called ferrite iron. Above Tt, in the γ phase, it has a face-centered cubic (or FCC) structure and is called austenite iron.

The transformation from ferrite to austenite can be very different from the austenite to ferrite transformation. The first one is an atom diffusion-based nucleation and growth transformation (the transformation first occurs simultaneously on preferential sites, creating nuclei which then grow into the surrounding untransformed phase). The second one can be either a nucleation and growth transformation again, or a diffusionless martensitic transformation. As defined in (2) : "a martensitic transformation involves a cooperative movement of a set of atoms across an interface causing a shape change and sound". As no diffusion is involved, there exists orientation relationships between the orientation of the original grain and the orientation of the formed crystals. The transformation from austenite to ferrite can be approximated by the Bain mechanism (although it is never observed in practice for pure iron).

The main objective is to exploit experimental data obtained on the device to identify the phase transition mechanisms, coupled with the temperature and plasticity effects. In particular, the ambition is to identify the transition point between the nucleation and growth transformation and the martensitic transformation during cooling of the sample. To do so, a sample with a small number of big grains (500μm to a millimeter in diameter) is observed during the transformation through digital image observation. The effect of the loading conditions on the initiation and progress of the phase transition will also be studied.

References :

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