TECHNICAL DEVELOPMENTS DEDICATED TO "IN SITU" TEMPERATURE ANALYSIS OF CRYSTALLOGRAPHIC TEXTURES BY NEUTRON DIFFRACTION

Ph. Gerber, J.P. Ambroise, M.H. Mathon

Laboratoire Léon Brillouin, UMR 12, CEA (DSM/DRECAM)-CNRS, CEA Saclay, 91191 Gif sur Yvette, France

The mechanical, physical and chemical properties of polycrystalline materials are strongly correlated to the distribution of crystallographic orientations. Controlling the crystallographic textures is crucial for the optimization of these properties and requires to precisely follow their development during each step of any industrial thermomechanical process. Neutron diffraction is particularly well-fitted with crystallographic textures analysis. This technique enables the achievement of good statistical measurements resulting from the large size of the investigated volume, consequence of the neutrons high penetration. The minority texture components are easily characterized, which makes it possible to follow the evolutions of the preferential components as of the first stages of their development.

A special furnace adapted to the texture measurement has been recently developed to allow "in situ" experiments on the 4-circles diffractometer 6T1. The furnace was built by Pyrox company and the technical improvements (heating elements, the coolant system, the regulation, ..) as its adaptation on the Euler's cradle were carried out by J.P. Ambroise.
The furnace (Fig. 1 and 2) is centred on the goniometer. It is constituted of heating elements achieved on molybdenum (1) that provides a maximum temperature of about 1000°C with a ramping rate of 300°C/min. The sample (2) is fixed on the sample mounting rod (3) with vanadium wires. The temperature is controlled by three thermocouples (4) displaced into the furnace (5), in close contact with the sample. Under normal conditions and without any readjustment, the temperature typically displays drifts that not exceed 1°C, even over several days. Vanadium foil is placed over the sample and acts as a heat shield. An aluminium sphere with a perfectly homogeneous thickness lower than 1 mm, constitutes the external envelope of the furnace and ensures its sealing. At the bottom of the device, a pump-out port for pumping high vaccum produces a controlled atmosphere of 10^{-3} Torr. Finally, a cooling water system minimizes heat transfers towards the Euler's cradle.

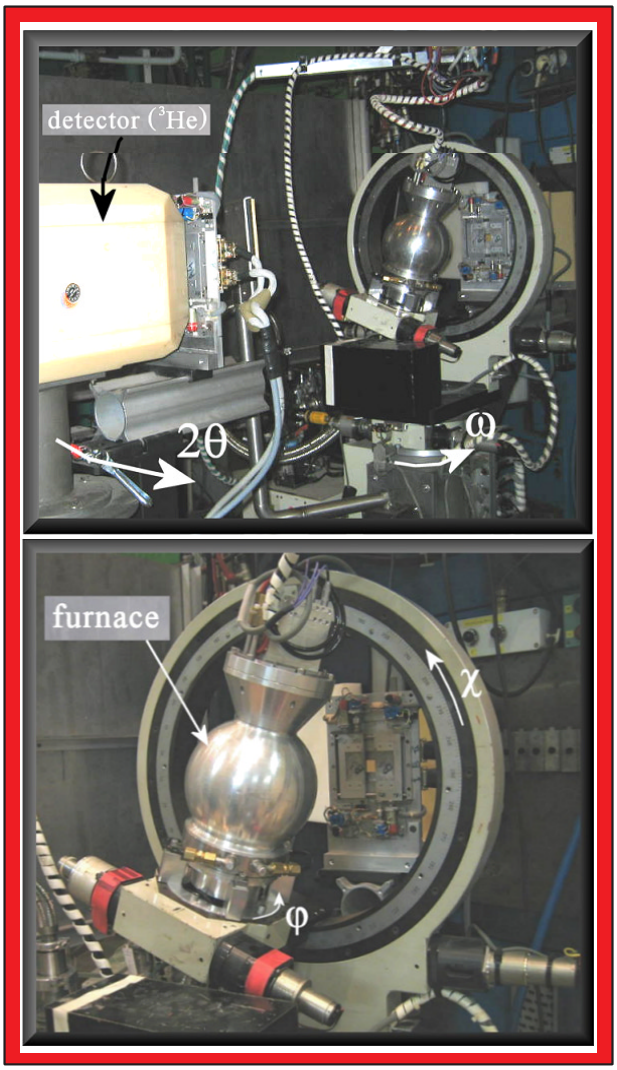

Figure 1 : Presentation of the furnace installed on the 4 circles 6T1 diffractometer

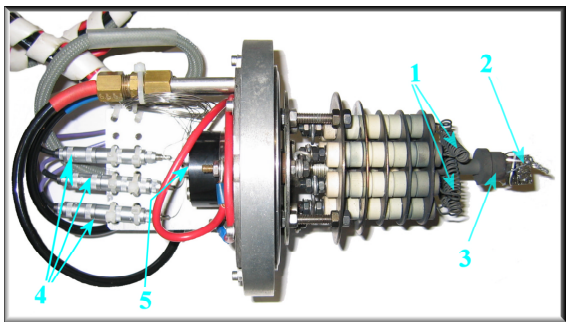

Figure 2. Upper and internal part of the furnace.

This new furnace equipment allows the study of the texture development during annealing process at high temperatures in plastically deformed materials and/or phase texture transformation. In order to analyze the texture evolution versus the annealing time, the experiment is performed following a specific pole relied to one major crystallographic orientation that appears or disappears during annealing. With a single-detector, recording time accuracy between 1 and 20 sec is possible. This time scale is compatible with the phenomena of recrystallization. One of the most important goal of recrystallization-of-deformed-materials science is to predict the overall kinetics, *i.e.* to forecast the time and temperature conditions under which the process will occur for a material. For example, static recrystallization has been analyzed in wire-drawn copper deformed for various strain levels (true strain between ε=0.73 to 2.84).

It has been determined in copper from neutron diffraction measurements, that the wire-drawn texture is composed of unbalanced <111> fiber (major orientation) and <100> fiber (minor one) [1,2]. According to the recrystallization textures analysis in copper wires, it appears that the deformation texture becomes sharper with increasing strain. During recrystallization, the <100> fiber is reinforced while the <111> fiber decreased drastically [2]. Hence, during "in situ" annealing at constant temperatures, the recrystallization kinetic can be followed through the decrease of the diffracted intensity at the {111} pole figure center. Note that the process could be followed through the increase of the diffracted intensity related to the <100> fiber and it should give the same results. For a given temperature, the reaction advancement factor *R(t)* of the recrystallization process is calculated by the relation:

$$R(t) = \frac{I(0) - I(t)}{I(0) - I(\infty)} \qquad (1)$$

with *I(0), I(t) and I(∞)* are diffracted intensities respectively at the initial deformed state, at a given time *t* and at the end of the recrystallization process. The Figures 3a and 3b present the *R(t)* evolution for two different deformation rates (ε=1.27 and 2.84) and different tempretures. They present a classical sigmoïdal shape with an incubation time followed by an increase of the recrystallization rate (linear region) and finally, a decrease of the recrystallization rate corresponding to the impingement of growing grains. Assuming that the growth of the grains is thermally activated, the activation energy associated to the recrystallization process, can be deduced from the results obtained at different annealing temperatures, according to Arrhenius equation:

$$a(t) = C.e^{\frac{-E_a}{kT}} \qquad (2)$$

E_a is the activation energy of the recrystallization process in J/mol. *k* is the Boltzmann constant equal to 8.314 J/K.mol and for a given temperature, *C* is a constant related to the incubation time.

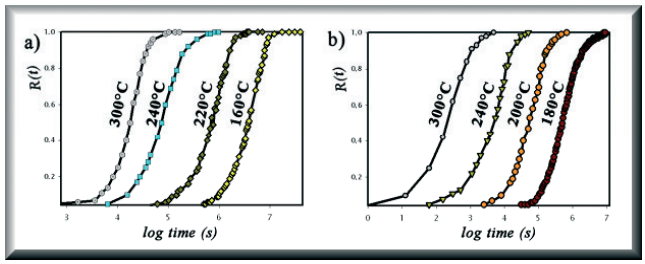

Figure 3 : Recrystallization kinetics of copper for two deformation rate a) ε=1.27. b) ε=2.84.

For the first time, the relation between *Ea* and the level of strain has been expressed. The figure 4 shows an exponential decay of the activation energy upon increasing strain. The fast decrease is rapidly slow down at the highest level of reduction and value of E_a≈45 KJ/mol is reached for a strain level equal and higher 2.29. The lower values of *Ea* obtained for higher deformation levels can be explained by the larger vacancies content that promotes easier boundary motion during recrystallization. These *Ea* values will be introduced in statistical models in order to predict the microstructure and texture evolutions during recrystallization.

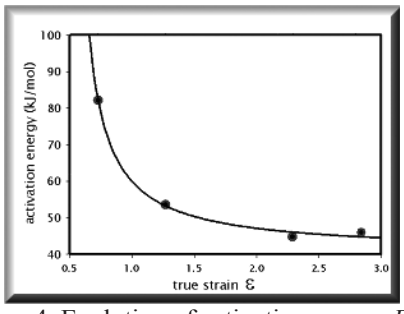

Figure 4. Evolution of activation energy E_a as function of the true strain.

References

[1] S. Jakani, M.H. Mathon, M. Benyoucef, Ph. Gerber, T. Baudin, C.H. de Novion, J. of Neutron Research, **126** (2004) 249.

[2] Ph. Gerber, S. Jakani, M.H. Mathon, T. Baudin, accepted for publication in *Mat. Science For*