



|  |   |                                      |
|--|---|--------------------------------------|
|  | <b>Experiment title:</b><br>In situ characterization of co precipitation kinetics of carbides and intermetallic phase | <b>Experiment number:</b><br>MA 1127 |
| <b>Beamline:</b><br>ID15b  | <b>Date of experiment:</b><br>from: 08/02/2011 to: 14/02/2011   | <b>Date of report:</b><br>14/01/2012 |
| <b>Shifts:</b><br>12   | <b>Local contact(s):</b> Thomas Buslaps   | <i>Received at ESRF:</i>             |
| <b>Names and affiliations of applicants (* indicates experimentalists):</b><br>Moukrane Dehmas SI2M IJL UMR CNRS 7198 NANCY<br>Guillaume Geandier SI2M IJL UMR CNRS 7198 NANCY<br>Mathieu Salib SI2M IJL UMR CNRS 7198 NANCY<br>Benoit Denand SI2M IJL UMR CNRS 7198 NANCY |   |                                      |

## Report:

The hardening in the maraging steel is due to a fine and homogeneous precipitation of intermetallic phase NiAl and  $M_2C$  carbide during aging. Chromium and molybdenum in this alloy are hardening elements as they contribute to the formation of these carbides. The grain growth at the austenitizing temperature is limited by the presence of vanadium carbide. Unlike conventional maraging steels, this steel contains no titanium, which has the effect to prevent the formation of titanium nitride during the elaboration. These nitrides, whose size can reach tens of microns, are the preferred sites of initiation of fatigue failure. Outlawing titanium contributes greatly to a significant gain in fatigue strength compared to the maraging 250. However, the fatigue of this steel is not yet satisfactory because of the formation of aluminum nitrides, with a specific morphology.

The recording of diffraction patterns during heat treatment allowed us to identify the present phases and their existence domain in temperature. We also quantified the mass fraction of the different phases (i.e. martensite/ferrite and austenite) as their lattice parameters. The evolutions of mass fractions of martensite/ferrite and austenite during heating and natural cooling after holding for 1 hour at 950°C are shown in Figures 1a and 1b. During heating, we observe that the austenitic transformation occurs from 600° C to 830°C. We detect the presence of aluminum nitride up to 730°C. Of course, this does not exclude the fact that they may be present in smaller quantities. We also noted on heating the appearance of new diffraction peaks in a temperature range between 620°C and 875°C. The identification of these diffraction peaks remains to be done. During the isothermal holding at 950 ° C, only the presence of austenite is detected. On cooling, there is a start of martensitic transformation in the vicinity of 210 ° C. The martensitic transformation progresses very quickly and its evolution is close to linearity with temperature. We also note that despite a rapid increase in the amount of martensitic phase, we

did not observe an exothermic nature of the transformation as in the case of maraging steels. The mass fraction of residual austenite after cooling is close to 20%.

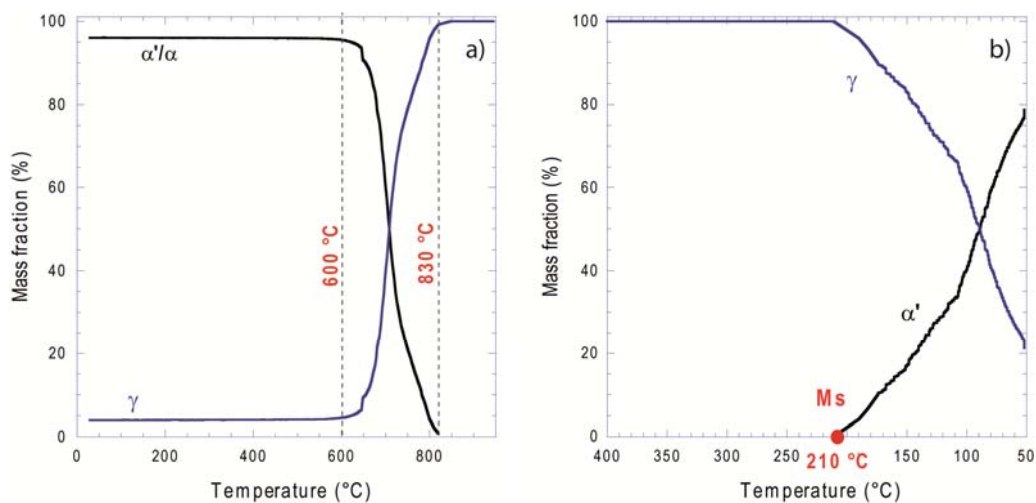


Figure 1: Evolution of martensite/ferrite and austenite during heat treatment from the initial state: a) heating at  $0.3^{\circ}\text{C}\cdot\text{s}^{-1}$  ; b) air cooling.

In parallel with the quantification of phase amounts, we measured the average lattice parameters of martensite/ferrite and austenite during the homogenization treatment. Their respective evolution reveals this complex evolution, which is not only due to thermal expansion, but also to mechanical and chemical evolution of the structure. Some of them are related to the austenitic transformation and release of interstitial solutes to form carbides or nitrides.

After homogenization treatment, the samples were maintained at  $-80^{\circ}\text{C}$  during 8 hours. The mass fraction of residual austenite is about 4%. It should be noted that we could not continuously monitor the evolution of phase fractions in the transition from the cold because the operation would have taken too long for the time runs out at the ESRF to change the experimental.

After isothermal holding at  $-80^{\circ}\text{C}$ , the samples were annealed at  $500^{\circ}\text{C}$ . No significant variation of mass fractions of martensite and retained austenite is observed in comparison to those obtained after passing through the cold. However, we identified the diffraction peaks associated with aluminum nitrides after 40 min isothermal holding at  $500^{\circ}\text{C}$ . Their intensity increases continuously with the isothermal holding time, suggesting an increase in the amount of aluminum nitride. SEM observations of the specimen after quenching and annealing confirm the presence of AlN with the faceted morphology. The size of these nitrides is between 1 and  $4\ \mu\text{m}$ . It is difficult to imagine the precipitation of aluminum nitride with a size as large during a heat treatment at "low temperature". Therefore, we assume that this is a growth of aluminum nitride existing after quenching and not detectable by high energy X-ray diffraction. Observations made by SEM on a sample treated with similar homogenization reveals indeed the presence of aluminum nitride after quenching. We did not exclude the presence of fine aluminum nitride during annealing. Finally, we also noted the appearance of other diffraction peaks during the annealing that we have not yet assigned to a phase. This is probably a carbide type  $\text{M}_2\text{C}$  but the task of identifying remains to be done.