ESRF	Experiment title: In situ characterization of bainitic and martensitic transformation under stress by high energy X-ray diffraction”	Experiment number: ME1188
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Shifts:	**Local contact(s)**: Gabriela Gonzales-Aviles	*Received at ESRF:*

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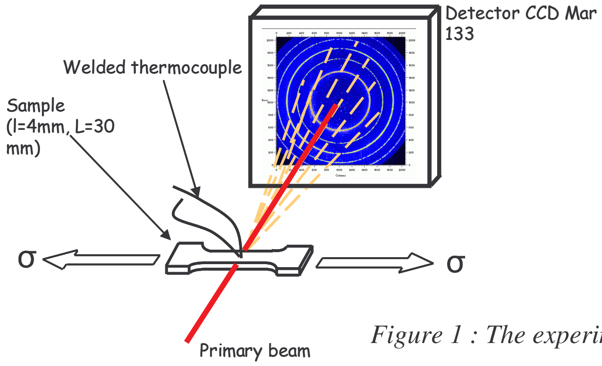
Report:

The aims of the experiments were to realize an in situ study of the effect of stresses on phase transformations, using high energy X-ray diffraction, and to further analyze the role of the applied stress on the behaviour of the alloy during the transformation. These experiments were performed thanks to the tensile machine from FAME38 (figure 1).



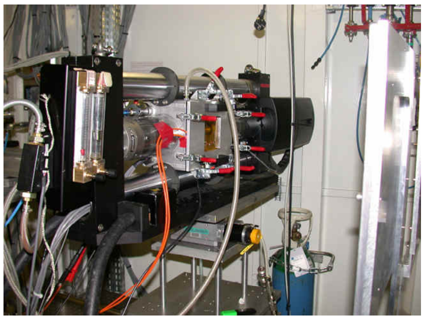


Figure 1 : The experimental setup

The specimen temperature was measured by a thermocouple spot welded on the surface of the specimen. As transformation kinetics is very rapid, a MAR 133 detector was used allowing a recording of the whole Debye Scherrer rings each 10 seconds. In order to have enough data during the transformation, cooling was controlled at a low cooling rate (-0.3°C/s). These conditions are different with the one used for the experiments performed in Nancy on the thermomechanical simulator. This choice has also an effect on the obtained data. The wavelength used was 0.13906 Å (E=89.906 keV). Different alloys were studied (a middle alloyed steel: 35NiCrMo16 and a Maraging steel (with a lower carbon content).

In all cases, the samples were heated up to the homogenisation (austenitization) temperature applying a small constant load of 0.0007 kN. The martensitic phase transformation is studied during continuous cooling. For that case, the load is increased to a constant value at a temperature above the Ms temperature (temperature at which the transformation begins). For the bainitic transformation, after austenitization the sample was cooled down to 320°C, and maintained 4h at that temperature. The load was increased and maintained constant at the beginning of the holding at 320°C.

First results

A circular integration on the Debye Scherrer rings has been performed in order to get the (I, 2θ°) diffraction patterns. Figure 2 shows the evolution of the diffraction patterns obtained during a treatment without applied stress on the middle alloyed steel. At the initial state, the ferritic phase (CC) is present. On heating, we observe the transformation α(CC)→γ(CFC). During the cooling, from 252°C, the martensitic phase transformation is well highlighted.

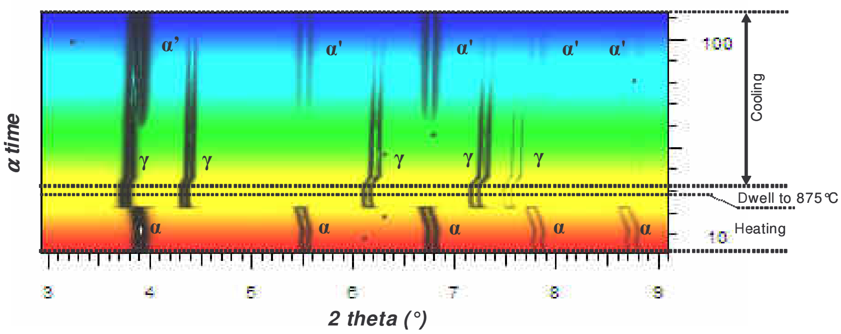


Figure 2 : 2D Representation of diffraction patterns obtained during a thermal cycle: heating to 875°C, dwell at this temperature, cooling at room temperature.

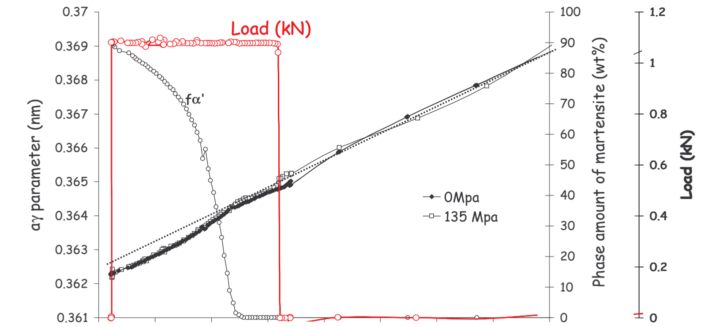


Figure 3:Evolution of phase amount of martensite (wt%) and mean cell parameter of austenite obtained during the cooling with and without an external applied stress.

From these diagrams, we could determine the transformation kinetics and the evolution of the mean cell parameter of each phase as a function of the temperature. As shown on figure 3, without applied stress, we obtain the Ms temperature (252°C) and the transformation kinetics. The in situ experiments allow to show that for a martensite fraction amount $f_{\alpha'} \geq 15$ wt%, the mean cell parameter of the parent phase a_γ deviates from the linearity. This change of behaviour is significant of a mean compression state in the austenite. The behaviour is similar for the Maraging steel.

When a stress is applied, the two alloys did not behave similarly. We observed an increase in the Ms temperature with increasing applied stresses for the Maraging steel. Moreover, the transformation kinetics is not simply shifted by the Ms increase as shown figure 4. When transformation progresses, the kinetics are overlapping. For the middle carbon steel (with a higher Ms temperature) no shift in the Ms the temperature was measured, and transformation kinetics are similar. This results is different from the one obtained by other techniques and has to be analysed further in regard of the low cooling rate. Indeed, analysis of cell parameters of martensite indicates clearly a self tempering of the martensite. The way this could affect the stress/kinetics relationship has to be studied.

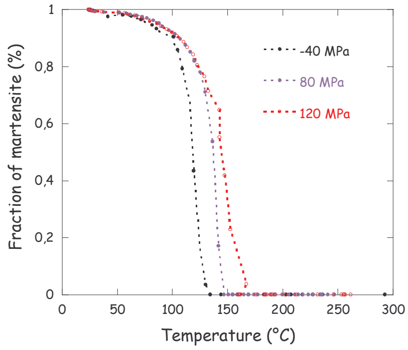


Figure 4: Evolution of phase amount of martensite versus temperature during treatment with different external applied stresses levels.

In order to analyse further the results, a partial integration of the Debye Sherrer rings in both direction (Ψ=0° and Ψ=90° where the direction Ψ=90° is the tensile direction) was performed. The angular sector of integration was equal to 1.8°. From these partials integrations, we have determined the interplanar spacing $(220)_\gamma$ during the treatment in both directions.

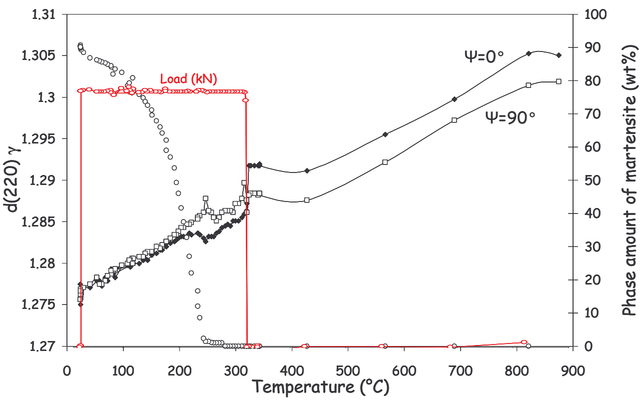


Figure 5 : Evolution of phase amount and interplanar spacing in the tensile direction and in the perperpendicular direction)

Between 875°C and 300°C, the distance $d(220)_\gamma$ obtained in the two directions are different. We relate this difference to the lower statistic (large austenitic grains size), perhaps the internal stress and the heterogeneity of temperature (due to the rapid cooling rate).

When the stress is applied (in the γ phase), the $d(220)_{\Psi=90°}$ value is larger than the $d(220)_{\Psi=0°}$ one, as expected for a tensile deformation in that direction.

When the transformation progresses the difference between $d(220)_{\Psi=90°}$ and $d(220)_{\Psi=0°}$ decreases, and both values are similar when martensite amount is equal to about 40%. As no differences can be observed from these partial integrations, we associated this behaviour to the increasing internal stresses developed during the transformation, which modify strongly the initial tensile stress state in the austenite.

Conclusion

We were able to realize the experiments allowing to study the martensitic phase transformation under an external stress, and characterize the phase evolutions by high energy X-ray diffraction during cooling with an applied tensile stress. The present results show that for the Maraging steel, the Ms temperature is increased with an applied stress. Results are similar to the literature. The kinetics is not simply shifted by the increase in the Ms temperature. First analysis of cell parameter evolutions have been done. Without an external applied stress, we are able to show that a mean compressive state is observed in the austenite at martensite amounts larger than 15%.

Additional analyses of the experimental results have to be done. Namely, to go further on the stresses analysis, it's necessary to reach the stress state in the phases by partial integration all along the diffraction rings, and to associate the experimental results with a micromechanical model.