Experimental Report

Proposal: **1-01-126** **Council:** 4/2012

Title: Stress ageing of a 15-5-PH martensitic stainless steels : SANS study of precipitation ans spinodal decomposition

This proposal is a new proposal

Researh Area: Materials

Main proposer: **DE GEUSER Frederic**

Experimental Team: DESCHAMPS Alexis
DE GEUSER Frederic
COUTURIER Laurent
FLAMENT Camille
SALLEZ Nicolas

Local Contact: DEWHURST Charles

Samples: Fe-15%Cr-5%Ni-3%Cu (wt%)

Instrument	Req. Days	All. Days	From	To
D11	3	2	27/02/2013	01/03/2013

Abstract:

The proposed experiment aims at obtaining a systematic characterization of the microstructural evolution during long-term ageing with or without external stress of the aerospace alloy 15-5PH martensitic steel. This long term ageing involves both spinodal decomposition linked to the high Cr content of the steel and evolution of the Cu nanoscale precipitates. These two contributions will be individually determined by using the difference in the magnetic and nuclear contrasts of these two contributions.

SANS characterization of spinodal decomposition and copper precipitation evolutions in a precipitation hardened martensitic stainless steel during long term ageing.

Introduction

The 15-5PH martensitic stainless steel is used for aeronautic applications as constitutive material for airplane pylons. Because of its proximity from engines it is subjected to ageing at moderate temperatures (~300°C). This results in microstructural changes giving rise to modifications of the mechanical properties. The characterization of the spinodal decomposition and its evolution which is the main responsible of properties modifications of the alloy is important to understand the transformation mechanism and to be able to predict the properties evolution.

Experimental

Near 60 ageing conditions were investigated (ageing times up to 6000h at 6 different temperatures in the range 290°C – 420°C) on beam line D11 under a magnetic field of 1.6T to saturate the magnetic domains. It was also planned to investigate samples aged under stress but for technical reasons we did not have such samples at hand.

The samples were cut from aged pieces in 10x15x3mm^3 parallelepipeds. Three different sample-to-detector distances (1.6m, 8m and 34m) were used in order to explore a wide q range (0.025nm^{-1} – 4.5nm^{-1}).

Results

This experiment on a large number of aged samples gave very satisfactory results, showing a clear evolution of the scattered intensity with ageing time (Figure 1). As expected, the reaction is faster for higher temperature.

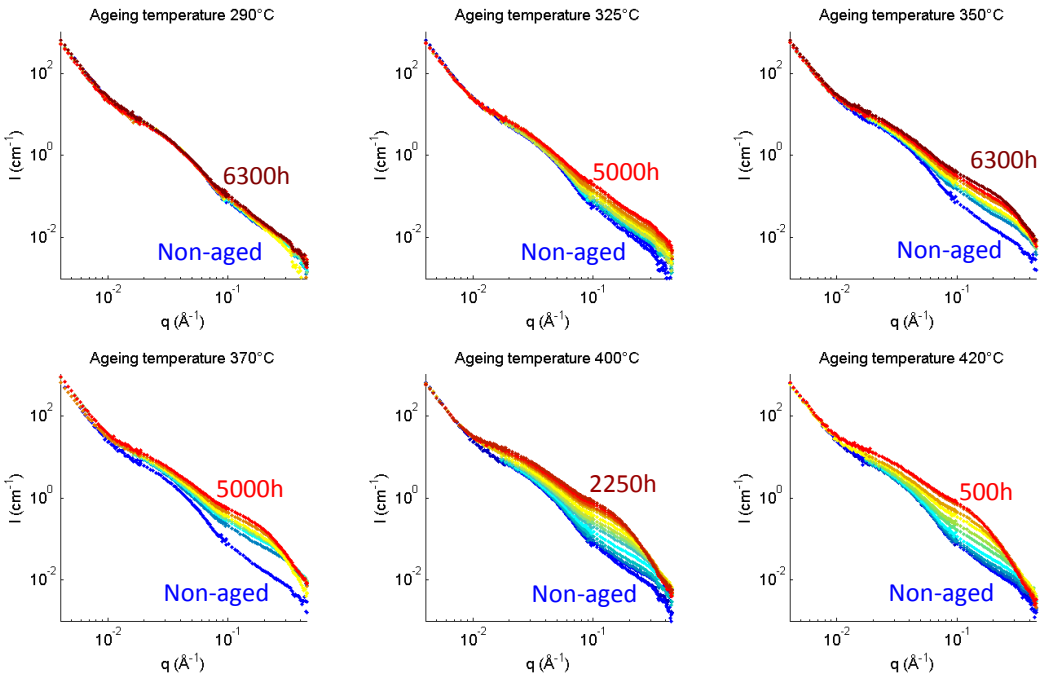

Figure 1: Evolution of the scattered intensity with ageing time (nuclear contrast)

In fitting the scattered intensity with 3 contributions, we were able to obtain the microstructural characteristics of the spinodal decomposition and of the copper precipitates as shown in Figure 2. As one can see on Figure 2, magnetic contrast enhances the relative contribution of the hardening precipitates to the total scattered intensity, making easier the separation of the contributions. In addition, nuclear contrast gives a higher relative contribution of spinodal decomposition to the signal than in SAXS experiments.

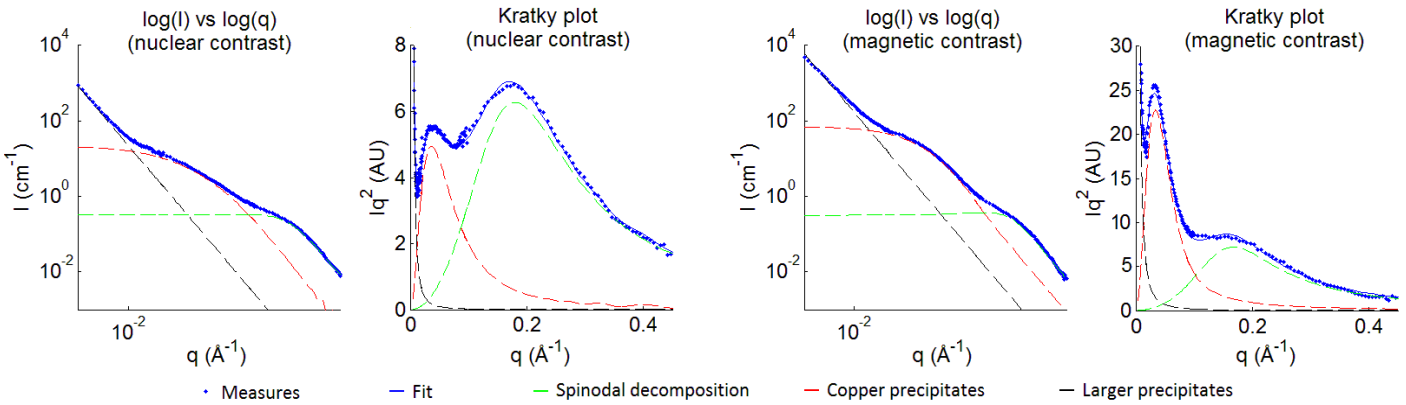

Figure 2: Fit of the scattered intensity, sum of 3 contributions: spinodal decomposition, copper precipitates and larger objects.

In considering data sets obtained for all the studied samples, we can obtain the kinetics of evolution of the different microstructural characteristics of spinodal decomposition and copper precipitation. We show in Figure 3 the evolution of the mean radius and of the corresponding computed integral intensity of the precipitates and the coherency length (characteristic length of the rich and poor zones) and the corresponding computed integral intensity of the spinodal decomposition.

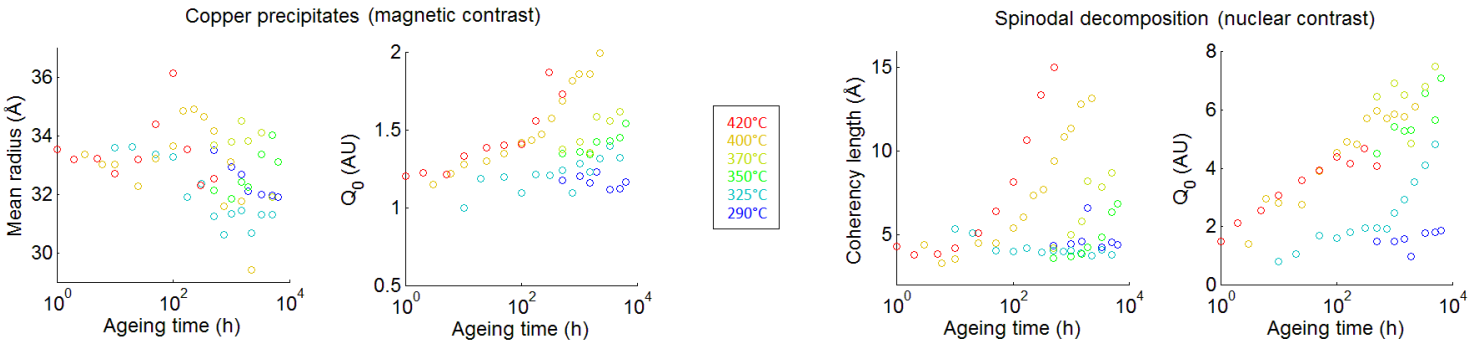

Figure 3: Evolution kinetics of mean radius and corresponding integrated intensity Q_0 of copper precipitates and of coherency length and corresponding integrated intensity Q_0 of spinodal decomposition (from the left to the right)

Conclusion

This experiment at the ILL brought a lot of important data concerning the spinodal decomposition kinetics and the evolution of the hardening precipitation in the 15-5PH steel. Together with data obtained from other techniques these data sets will soon give rise to publications.