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SANS study of structure degradation in long time thermally exposed wrought nickel base superalloy

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A study on the structural stability of the wrought nickel base superalloy EI698 VD exposed for a long time under thermal conditions was performed in this work The presented Small-Angle Neutron Scattering (SANS) experiment was a part of a broader investigation of a long-time structural stability of wrought nickel base superalloy EI698VD and its effect on the subsequent creep characteristics. In order to study the structural stability, the specimens were thermally exposed to over 25000 hours at two temperatures, 430°C and 650°C, prior the creep. The creep deformation behavior of alloy exposed to different period was then evaluated. The structure characteristics changes including γ’ precipitate morphology, size and grain boundary carbide precipitation were investigated using scanning and transmission electron microscopy analysing techniques. While these results showed no important microstructural change, the rupture life of the alloy decreased with the increasing exposure time. The SANS experiment was a crucial part of a broader investigation of a long time structural stability of superalloy and has revealed significant changes of the morphology of γ’ precipitates after the thermal exposure.

1. INTRODUCTION

Excellent mechanical properties of nickel base superalloys results from their two-phase structure, where the coherent γ’ precipitates are embedded in a matrix formed by a γ phase. One of the most important characteristics of these materials is their creep resistance. In the polycrystalline superalloy EI698VD large differences in the creep were observed when the samples were previously exposed to the isothermal loading for a long time at relatively low temperatures [1]. The differences in the creep behaviour were not practically reflected in the structural characteristics changes which were investigated using TEM and SEM analyzing techniques. The rather more significant structual changes than those observed were expected

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to clarify the creep behaviour.Small Angle Neutron Scattering (SANS) method has been proved to contribute substantially to the investigation of the microstructure of Ni-base superalloys. Important is its contribution in the field of determination of the γ'-phase morphology [2-4]. The SANS is caused by fluctuations of scattering length density ρ(r) due to compositional and/or structural inhomogenities which give rise to the scattering contrast. Generally, the SANS technique concerns the characterization of inhomogeneities in solids in the range 0Å - 5µm.

The SANS experiment was carried out in order to evaluate the microstructural changes of thermally exposed superalloy EI 698VD, and to contribute on the understanding of the discrepancy between creep experiments and TEM structural observations

2. EXPERIMENTAL

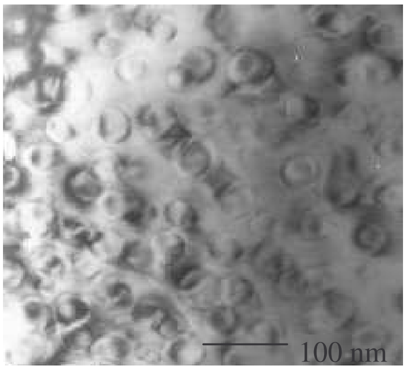

Fig.1. TEM micrograph of γ' morphology in initial alloy

Experiments were carried out on commercial nickel base superalloy EI 698 VD. The chemical composition of the alloy (in weight %) is presented in Table 1. The nickel base superalloy EI 698 VD is a solution and precipitation strengthened two phase alloy. The alloy was subjected to the following three stage heat treatment: a) solution annealing at 1100°C / 8 h, air cooling; b) precipitation aging at 1000°C / 4 h, air cooling; c) precipitation aging at 775°C / 16 h, air cooling.

The microstructure of the heat treated alloy before thermal exposure in initial material is displayed in Fig. 1.

Table 1.
Chemical composition of EI 698 VD superalloy in wt. %.

C	Cr	Al	Ti	Mo	Fe	Nb	Mn	Ni
0.08	13 -16	1.3– 1.7	2.3-2.7	2.8-3.2	max. 2.0	1.8-2.2	max.0.4	balance

The bars, which were used to machine the creep specimens after the thermal exposure, were exposed for 2000, 5000, 8000, 10000, and 25000 hours at two different temperatures, 430°C and 650°C. The exposure temperatures were chosen on the basis of gas turbine manufacturer requirements and they resulted from temperature measurement records on the cross section of a crankshaft at the time of turbine loading regime.

The creep tests were carried out using exposed specimens. A maximum loading force of 19.62 kN was applied which corresponded to 706 MPa. The crep testing temperature was of 650°C. The absolute elongation of each specimen was measured by an extensometer. The creep deformation ε versus time were frequently logged. The creep resistance of the exposed alloy was evaluated through the elongation at fracture ε and the time to fracture t_f.

Samples of approximately 1.5 mm thickness were cut from the head of crept specimens, to perform SANS diffraction measurements. The measurements were performed at room temperature at the V4 SANS facility in the Berlin Neutron Scattering Center (BENSC) at the HMI Berlin. The data were collected by a 2D position sensitive detector with a sample-to-detector distance equal to 16 m and $\lambda = 12.0$ Å. This geometry was selected in order to enable the observation of the large γ' precipitates and distances between them. The collected data covered the scattering vector magnitude range that is suitable for determining particle sizes between approximately 150 Å and 2000 Å. The measured isotropic scattering curves were corrected for the background and set to the absolute scale [5].

3. RESULTS AND DISCUSSION

The results of the microstructural analysis of the thermally exposed specimens confirmed that the thermal exposure of 10000 hours at 430°C and 650°C was sufficient to promote additional precipitation of the carbide phase locally along sections of grain boundaries. No other microstructural changes, such as change in the size of γ' particles, a change of their interfacial coherency with the matrix, or changes in their volume fraction, were observed. The exposure to over 10000 hours produced no additional microstructural changes. Only further carbide precipitation at grain boundaries and also carbide precipitation along twin boundaries was observed after the exposure of 25000 hours at both temperatures.

The creep results from differently thermally exposed alloy were reported in [1]. The creep deformation of specimens showed that the thermal exposure reduced the rupture life of all the specimens. It was possible to state that the longer was the exposure time the more detrimental was its effect on the recorded creep lifetime.

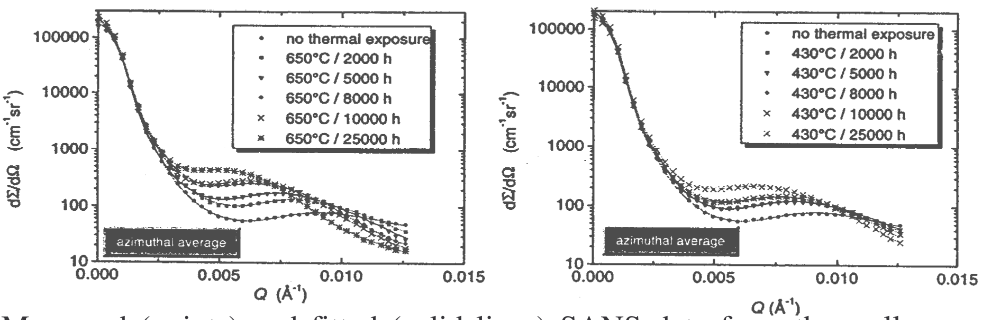

Fig. 2. Measured (points) and fitted (solid lines) SANS data from thermally exposed superalloy at both temperatures

The conducted SANS measurements resulted in the isotropic scattering curves. The resulting differential cross-sections dΣ/dΩ are displayed in Fig. 2 in logarithmic scale for different time of the thermal exposure. Each curve exibits an interparticle-interference maximum that indicates to a certain extent ordered precipitate microstructure. The curves were evaluated by a special procedure [6] suited for anisotropic data evaluation. The model used for this Transformed Model Fitting evaluation method [7] was a size distribution of cuboids forming a 3D binary map in the real-space. Both size and distance distributions were locally randomly smeared in order to obtain realistic approximation of the precipitate morphology. A long-range size distribution was included as well. The shape parameter was fixed to simulate a rather spherical form which can be deducted from micrograph in Fig. 1.

The SANS experiments revealed significant changes to the γ' morphology and distribution which became more pronounced as the time of thermal exposure increased. Some of the model distributions corresponding to the measured data for the exposed samples at temperature of 650°C are displayed in Fig. 3. The global change of the microstructure is evident. These changes can be considered as evidence of the creep properties degradation with respect to the applied period of alloy thermal exposure.

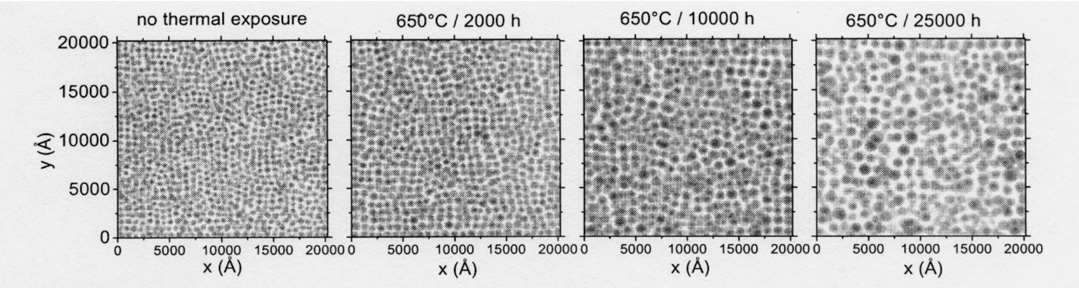

Fig. 3. Some of the real-space model distributions resulting from the SANS data for temperature of thermal exposure of 650°C

4. CONCLUSIONS

The following conclusions can be drawn from investigation:

- Gamma prime precipitate distribution models are optimally correspondig to the measured data for differently exposed samples.
- The resulting dependencies make evident that both the γ' size and the interparticle distance increase in the bulk of the material γ' with increasing thermal exposure. As expected, the increase is steeper for the samples exposed at temperature of 650°C. The distributions of precipitatets are rather broad and it can explain why this increase is not observed when using experimental techniques providing local information only.

The revealed evolution of microstructure can affect strongly the creep properties and is most probably the reason of large differences of the time to fracture and of the deformation ability of superalloy in dependence on the previous thermal exposure at relatively low temperature.

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