DETERMINATION OF CREEP STRAIN IN ALLOY IN 100

BY SMALL ANGLE NEUTRON SCATTERING

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Abstract

Specimens of alloy IN 100 creep tested at 950 °C in the load range of 65 to 110 MPa to a range of stepwise increasing creep strains were measured by small angle neutron scattering (SANS). A new procedure based on the anisotropic scattering signal of the crept specimens parallel and perpendicular to the load axis was used to evaluate the SANS data.

Two parameters were derived from the SANS measurements which showed a good correlation with the creep strain of the specimens, nearly independent of the load applied during creep testing. These parameters allowed specimens with strains lower than 1.0 % from those with higher strains to be distinguished. Based on the findings it is possible to determine the local creep strain in creep tested specimens.

This study encourages to verify SANS measurements on turbine blades with different service times in aircraft engines. The objective is the non-destructive measurement of local creep strain in order to get information on the residual life-time of turbine blades with unknown load history.

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Introduction

The service life of gas turbine blades is in part determined by changes in overall geometry, such as integral creep elongation, and by local strain limits. To date, the measurement of the local creep strain in blades by non-destructive techniques during turbine maintenance is not possible. In addition, service life calculation of turbine blades is a formal procedure only, due to incomplete knowledge about the load history during service.
Several attempts have been made to use small-angle neutron scattering (SANS) to determine residual life-time of turbine blades (1-4). In this paper, a new approach is made to correlate SANS results with the creep strain in nickel-base superalloy components.

Experimental

Material

IN 100 (Vakumelt ATS 391-G, Thyssen Guss Co.)
HIP: 4 h, 140 MPa Argon, 1228°C. Nominal composition (wt.-%):
15.0Co; 9.5Cr; 5.5Al; 4.8Ti; 3.0Mo; 1.0V; 0.18C; bal. Ni.

Creep tests

As the basis for correlating SANS data with creep strain, a series of specimens of IN 100 was creep-tested in helium at 950 °C and stresses of 65 to 240 MPa. Specimens were removed at stepwise increasing creep strains up to fracture strain.

High temperature heat treatment (HTT)

HTT was carried out at 950 °C for the same duration as the loading of the creep specimens. Having for each creep tested specimen a corresponding thermally exposed specimen, enables the influence of temperature and load on the microstructural variations to be distinguished.

Experimental techniques

The methods used for microstructural investigations were small angle neutron scattering (SANS) and for reference optical microscopy, scanning (SEM) and transmission (TEM) electron microscopy.

For scanning electron microscopy investigations a Leitz-AMR 1600 T system was used. The transmission electron microscopy studies were performed with a JEOL JEM 200CX. Both systems were equipped with an EDX unit of Tracor Northern.

The SANS measurements were performed with the SANS-2 facility at the FRG1 reactor of the Forschungszentrum (GKSS) Geesthacht using neutrons with $\lambda = 0.7$ nm, moderated by a cold source. The sample-to-detector distance range was 2 to 20 m, corresponding to scattering vectors of $0.02 \leq q \leq 1$ nm^{-1}, with $q = 4\pi\lambda^{-1}\sin\theta$. 2θ is the full scattering angle.
The neutron beam cross section was 9 x 9 mm^2. The thickness of the specimens ranged from 2.5 to 5 mm, thus providing representative quantitative data for an investigated volume of about 200 to 400 mm^3. Scattered intensities were recorded by a two-dimensional position sensitive detector. The intensities parallel and perpendicular to the load axis of the creep-tested specimens were calculated from the two-dimensional data set.
Subsequently the intensities were corrected for background and detector efficiency and then normalized to absolute unit of the differential cross-section by using a lupolene standard, precalibrated with vanadium.

Results and discussion

Creep tests

Creep rupture strength measurements at 950 °C are shown in Fig. 1a. The results fit well with literature data in the same temperature range.
Examples for the creep curves at 950 °C and 110 MPa are plotted in Fig. 1b. Except for two specimens not shown here, the creep behaviour of the different specimens is similar. A corresponding diagram for the strain rate versus time is plotted in Fig. 1c.

Microstructural investigations by optical and electron microscopy

Fig. 2 shows the typical morphology of superalloys (5-8) with a high primary γ' content. The shapes of the γ' precipitates in the as-received material (a, top) are somewhat irregular compared to the cuboidal particles after annealing for 160 h at 965 °C (b, middle). The material creep tested at 140 MPa, 965 °C for 160 h to rupture developed a structure of extended plates (rafting) (c, bottom).
The coarsening of the primary γ' precipitates, referred to as size range 3, with heat treatment is described by the two size histograms in Fig. 3 for (a) the as-received (hot isostatic pressed) material and (b) after 160 h at 965 °C. The mean values of the distributions determined by quantitative image analysis of the SEM micrographs (shown in in Fig. 2a and b) are shifted from about 430 nm to 685 nm. After 1600 h at 950 °C, we found a mean diameter of 1180 nm. The quantification of the morphology of the creep tested specimens in terms of size, shape and separation distance of the γ' phase after the transition from the cuboidal to the plate-like shape (rafting) is difficult.

A qualitative description of the shape transition in the specimens creep tested at 110 MPa and 950 °C is given in Fig. 4 as a function of creep strain. Specimens with strains in the range of 0.2 to 0.5 % show a transition from cuboidal to plate-like primary precipitates; at about 1 % strain the full plate structure is developed. In addition to the crept specimens, Fig. 4 shows the morphology changes of specimens heat treated at the same temperature of 950 °C (upper time scale) for the same durations as the loadings of the creep tested specimens. Only after a long annealing time of about 1500 h which corresponds to a creep duration close to rupture of the crept specimens, the tendency to form a type of the plate-like structure was found in the heat treated specimens.

During furnace or air cooling from service to room temperature, secondary γ' precipitates are formed in the γ phase matrix between the primary γ' precipitates (size 3) as in most γ'-hardened alloys. A bimodal size distribution of these secondary γ' precipitates with mean diameters in the range of 10 to 15 nm (size 1) and 25 to 45 nm (size 2) was found for our experimental conditions. The precipitates of the size ranges 1 and 2 are shown in the TEM dark field micrographs of Fig 6 (9).

The nucleation and growth of the secondary γ' precipitates in the supersaturated matrix during cooling is strongly influenced by the concentration gradients of the precipitate constituents, by the diffusion coefficients of Al and Ti in Ni as well as by the cooling rate. Dislocations (Fig. 7) at the γ/γ' interface may influence the diffusion and therefore may lead to an creep-enhanced coarsening of particles.

The mean diameter of the very fine secondary γ' precipitates (size range 1) hardly varies with creep strain, but there are indications for a change in number density of these precipitates with increasing strain. In contrast to the very fine particles, the mean diameter of size range 2 in-

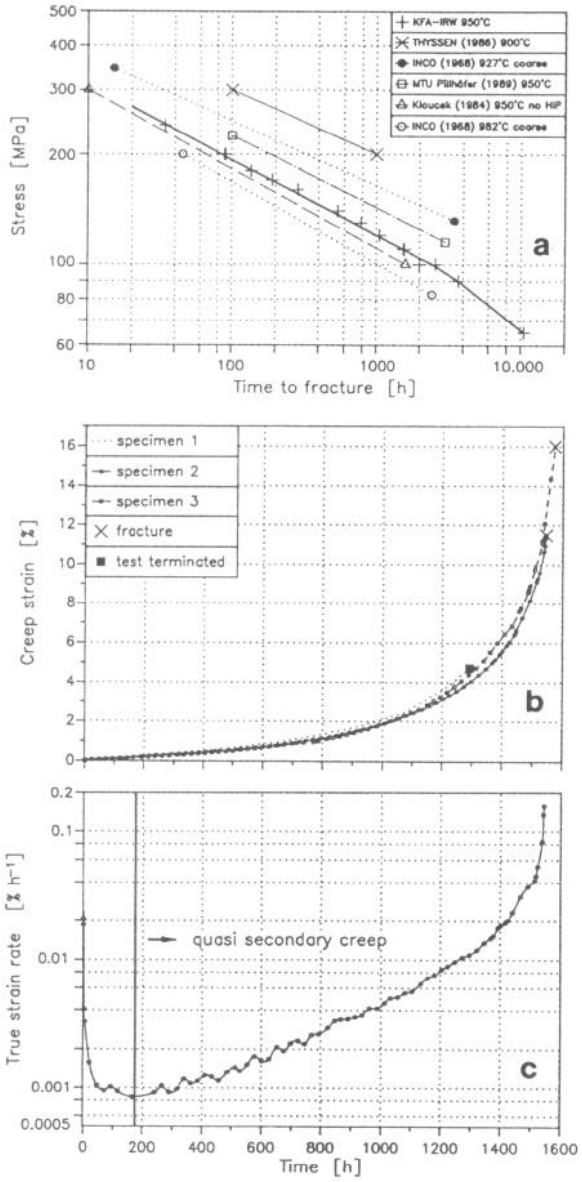

Fig. 1 a) creep rupture strength diagram of alloy IN 100 in the temperature range of 900 to 980 °C
b) creep curves at 110 MPa, 950 °C.
c) strain rate-time diagram at 110 MPa, 950 °C.

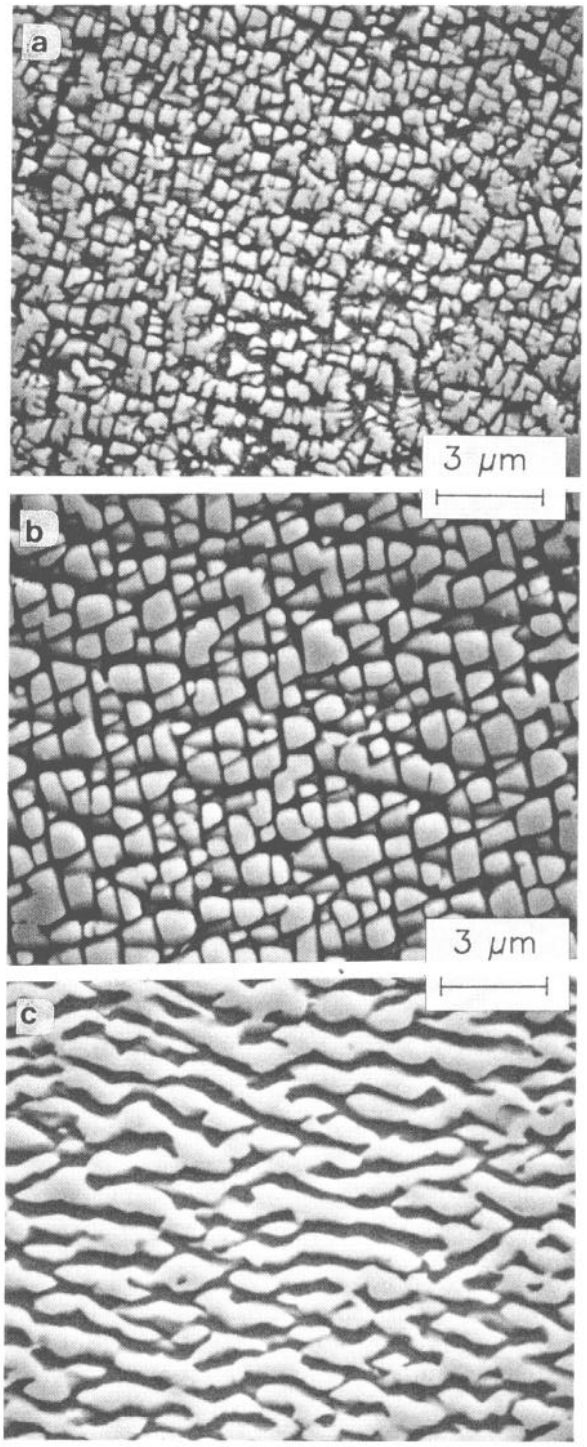

Fig. 2 SEM-micrographs of alloy IN 100
a) as-received
b) heat treated (160 h, 965 °C)
c) creep tested (140 MPa, 160 h, 965 °C, rupture)

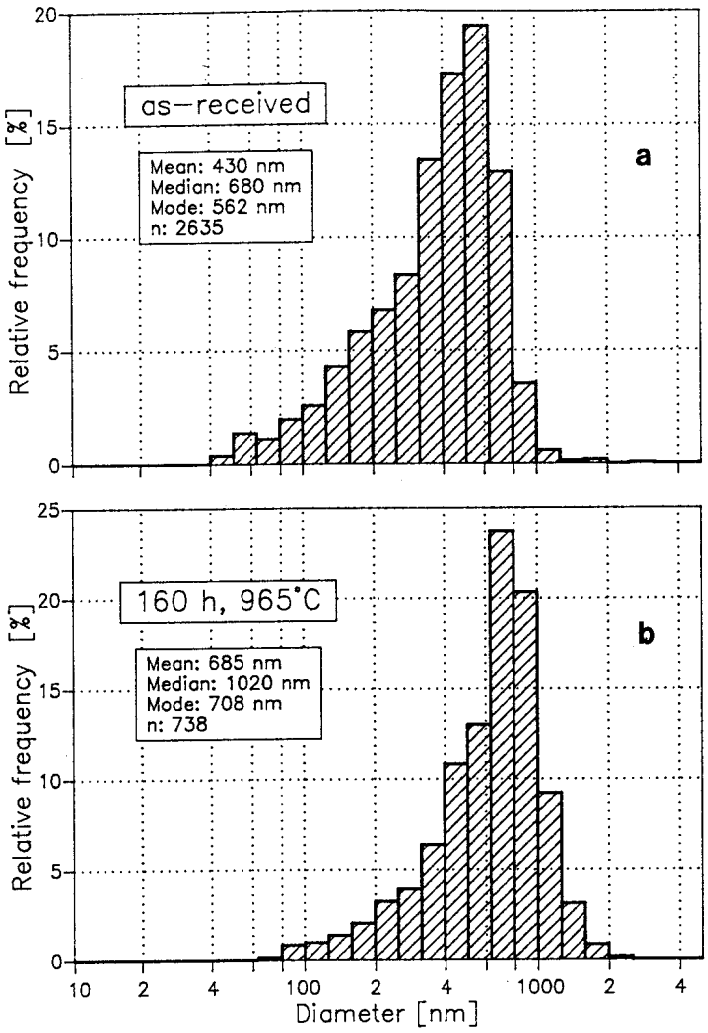

Fig. 3 Size histograms of primary γ' precipitates (size 3) determined by quantitative image analysis of SEM micrographs in Fig. 2
a) as-received
b) heat treated (160 h, 965 °C)

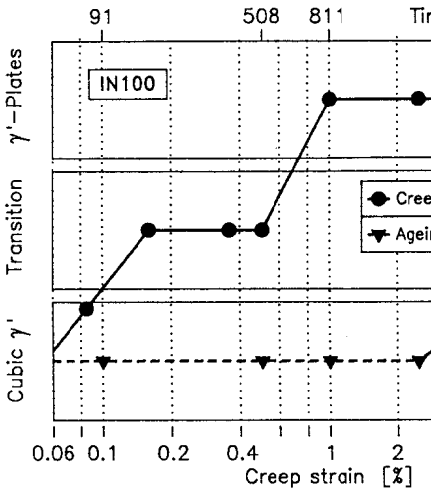

Fig. 4 Qualitative description of shape tra
tates (size 3) from cuboidal to pla
increasing creep strain or annealing

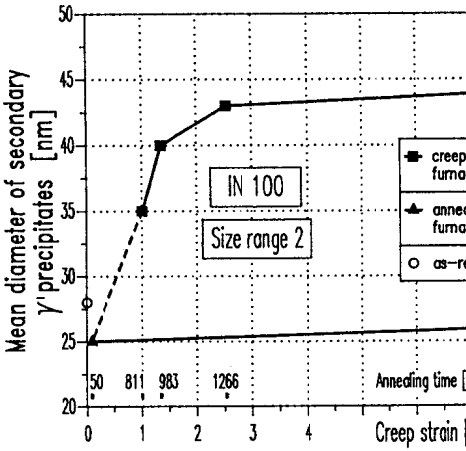

Fig. 5 Size variation of secondary γ' pre
increasing creep strain or annealing

creases with increasing creep strain up to a maximum value of about 45 nm at 2.5 % strain. The coarsening of these secondary precipitates is shown in Fig. 5 and compared to specimens heat treated at the same temperature of 950 °C (upper time scale) (9). Whereas annealing did not change the particle size, the influence of creep on the mean particle diameter of size range 2 is pronounced.
There is a small difference in precipitate size for the as-received specimen (28 nm) and for the specimen annealed for 50 h at 950 °C (25 nm). This may be due to the uncertainty of the measurements as well as to differences in the cooling conditions of the as-received specimen in a large furnace after HIP-procedure compared to the furnace cooling of the other specimens in the creep test facilities.

SANS measurements of heat treated or creep tested specimens

An example for scattering curves of the creep tested alloy IN 100 is shown in Fig. 8 for the as-received material a specimen creep tested to 7.6 % strain. Even in this logarithmic plot with a dynamic range of six orders of magnitude, the variations of the scattering curves with creep can be seen.

The high volume content and the quasi-periodical morphology of the primary γ' precipitates in the alloy complicate the classical evaluation of SANS curves in terms of particle size distributions and their volume fractions. A different approach for SANS intensity interpretation was therefore used which leads to a correlation of SANS intensity to local creep strain in creep tested specimens.

The procedures for the evaluation of the SANS data are:
- For each scattering vector q the ratio of the two SANS intensities parallel and perpendicular to the stress direction (load axis) during creep testing was calculated. The resulting curves for the quotient of the intensities Q(q) of the specimens with 1.0 and 7.6 % strain are plotted in Fig. 9. The Q(q) function for the as-received material is a horizontal line, as no anisotropic scattering was observed for this material. In contrast, the anisotropic structural variations in the creep tested specimens lead to changes in the Q(q) functions with strain. Fig. 9 shows that the function Q(q) has a relative maximum $Q_{max}(q_{max})$ in the q range of 0.1 to 1.0 nm^{-1}.
- The plot of q_{max} of each creep tested specimen as a function of the individual creep strain leads to the relationship shown in Fig. 10. In this diagram the data of specimens creep tested under different and in some cases changing loads in the range of 65 to 110 MPa indicate, that the correlation function between SANS results and the creep strains of the specimens is nearly independent of the load applied during the creep tests. To complement the data set in the strain range of 0.5 to 3 %, additional measurements will be performed on specimens creep tested at 65 and 90 MPa.
- Fig. 11 shows the plot of Q_{max} as a function of q_{max} for the same set of creep test specimens as in Fig. 10. This "SANS map" allows specimens with strains lower than 1.0 % and those with higher strains to be distinguished.

The evaluation of SANS measurements on the basis of the ratio of the scattered intensities parallel and perpendicular to the load axis, reflecting the creep induced anisotropic microstructural changes in the specimen, has a number of advantages.
1) the calibration of absolute intensity of the primary neutron beam is not needed.
2) the neutron absorption of the individual specimen is of no concern and allows easy measurements of specimens with uneven surfaces or varying thickness (e.g. turbine blades).
3) no reference specimens (as the as-received material) are needed.

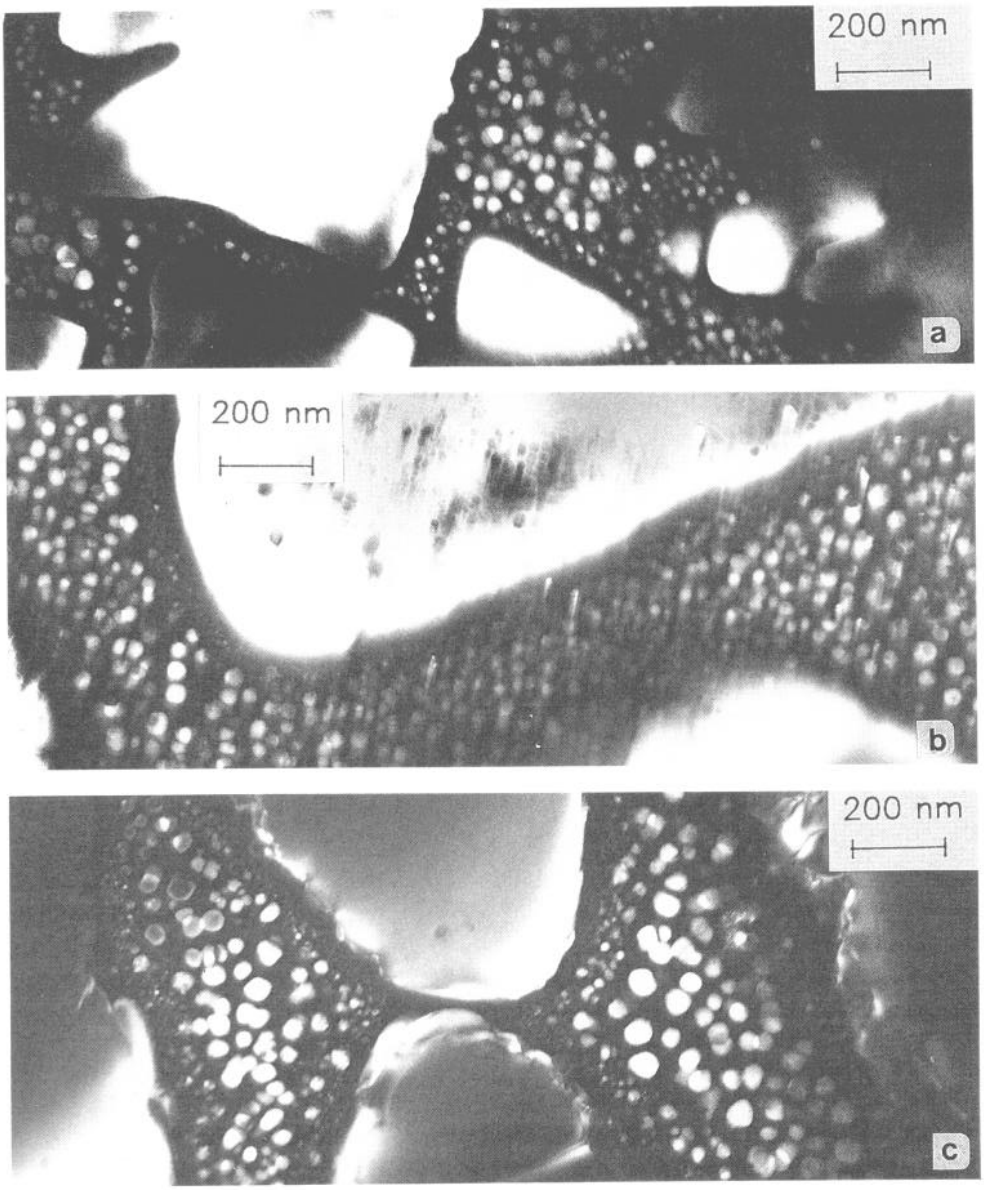

Fig. 6 TEM micrographs (dark field) of secondary γ' precipitates (size ranges 1 and 2) (9)
a) as-received
b) heat treated (1600 h, 950 °C)
c) creep tested (110 MPa, 2.5 %, 950 °C)

Fig. 7 TEM micrographs (bright field) of dislocation networks at the γ/γ' interface in an creep tested specimen (110 MPa, 7.6 %, 950 °C)

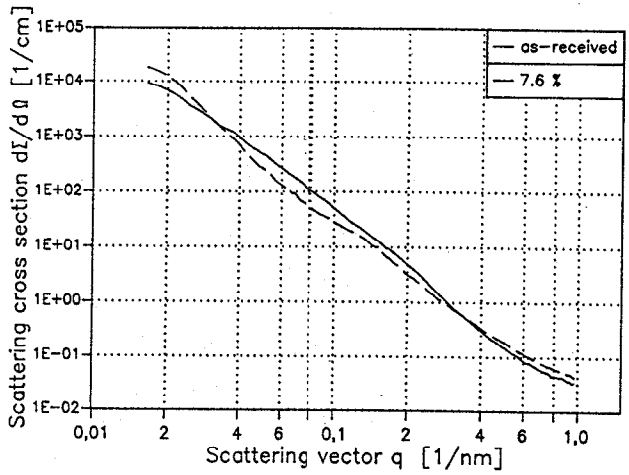

Fig. 8 Scattering curves $d\Sigma/d\Omega = f(q)$ of alloy IN 100 (as-received and creep tested to 7.6 % creep strain)

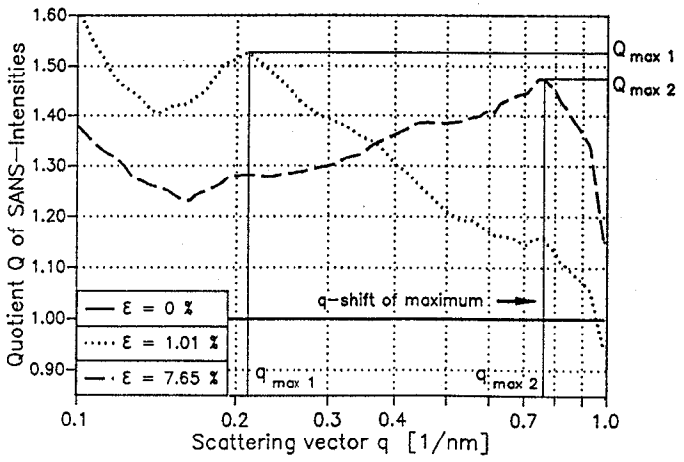

Fig. 9 A plot of the quotient Q of the SANS intensities parallel and perpendicular to the creep load axis allows the determination of q_{max} and $Q_{max}(q_{max})$. With increasing creep strain q_{max} is shifted to higher q values.

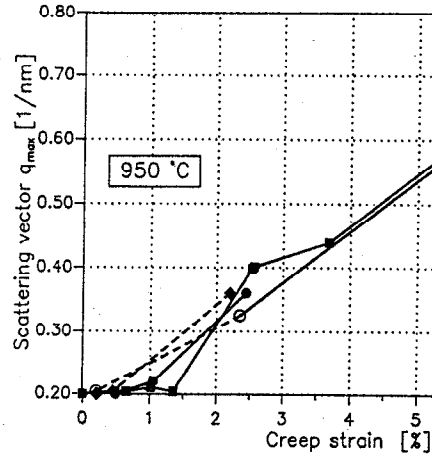

Fig. 10 Correlation of strain in creep tes
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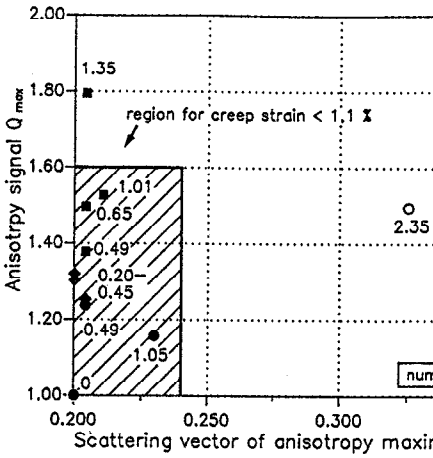

Fig. 11 A plot of Q_{max} as a function o
strains lower than 1 % and thos
tinguished. Q_{max} is related to the
creep tested specimens.

During SANS investigation a component can be scanned to determine the variation of local creep strain. For this purpose the primary neutron beam size can be reduced, depending on the measuring time allotted. On the other hand a specimen may be measured only at the highest loaded area.

The load history of a component (i.e. a turbine blade of an aircraft engine) is often not well known. It is therefore important for residual life-time determinations that the property measured non-destructively shows an unambiguous relation to life-time consumption and is not dependent on temperature or load applied.

Conclusions

The results of our study so far indicate that the relationship found between SANS measurements and creep strain as an indicator of consumed life time is independent of the creep load in the range of 65 to 110 MPa. The influence of creep temperature on the SANS intensity-creep strain relation will be studied using a series of specimens creep tested at 900 °C in addition to the 950 °C specimens.

Another investigation in progress deals with the relationship of creep strain, temperature and cooling rate with the formation of the secondary γ' precipitates as well as with the microstructural identification of the SANS scattering centres.

Finally, SANS measurements of blades with different service times are planned.

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