ESRF	**Experiment title:** **Characterisation of creep pores in Ni-Base alloys**	**Experiment number**: Me 375
Beamline: ID19	**Date of experiment**: from: 17.4.2001 to:20.4.2001	**Date of report**: 27.4.2001
Shifts: 9	**Local contact(s)**: Xavier Thibault	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. Dr. Walter Reimers

Dr. Anke Pyzalla*

Dr. Thomas Link*

Address for all 3 persons:

Institute for material science and technology

Technical University Berlin, Sekr. BH18, Ernst-Reuter-Platz 1,

D-10587 Berlin

Report:

Aims:

Single crystals of Nickel base superalloys show micro pores, resulting from solidification, heat treatment and creep deformation. The aims were to investigate methodical questions

- Checking,whether synchrotron tomografy can detect pores smaller than 10 µm in high density material.
- Introducing of a method for destruction free characterisation of micro pores

and metall physical questions

- Comparing the porosity in fully heat treated superalloys of different generations
- Measuring the change of porosity during creep at 1100°C for a certain material

Materials and method:

An overview of the investigated specimens gives table 1. The standard heat treated (SHT) and creep deformed specimens have cylindrical shape with the cylinder axis close to the [001] crystal axis, which is the growth direction during dendritic solidifacation. For the measurements we cut needles with about 14mm length in [001] direction and 0.5mm length in [100] and [010] direction. After cutting the needles were polished with perchloric acid to remove sharp corners. A needle was mounted with a special holder on a goniometer installed on the specimen table, the needle axis aligned along the z-rotation axis of the diffractometer. A FreLoN-camera with a resolution of 0.7µm/pixel was used and a beam energy of 50 keV. The scanned specimen volume is about $0.5*0.5*1mm^3$. We took about 35 images, one image has about 1.6 Gb after reduction of the grey scale on 8 bit.

Specimens					Results	
Material	Heat treatment	Temp.	Stress	Time	Pore density	Pore shape
SRR99	SHT	undeformed			Low to middle	round
CMSX-4	SHT				middle	round
CMSX-6	SHT				low	round
CMSX-10	SHT				high	round, very big
CMSX-4	SHT	1100°C	120 MPa	25 h	middle	Big round
				150 h	middle	Small square and big round
				292 h	high	square
			105 MPa	692 h	high	big square
			117 MPa	392 h fracture	high	square
			135 MPa	149 h	Middle to high	square

Table 1. Parameters of the investigated superalloys

Results

Results are taken from x-y cross sections through the tomography picture by the FASTTOMO software. From each of the 10 specimens we scanned 3 needles, for each scan we looked at about 6 cross sections. The qualitative results are listed in table 1. Figure 1 gives an optical impression of the findings.

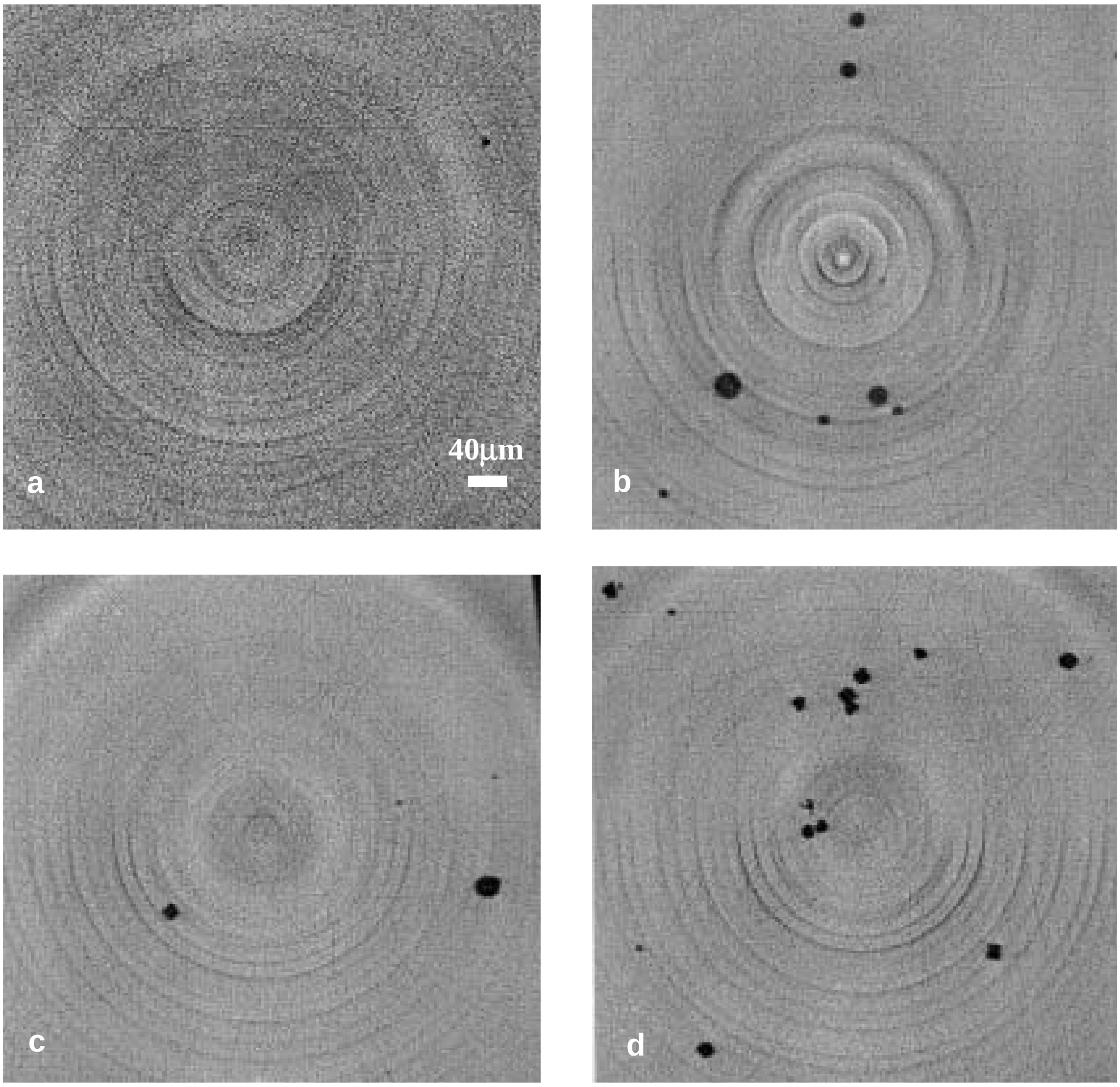

Fig.1. Tomography images of undeformed and creep deformed superalloys
a) CMSX-6, SHT, b) CMSX-10, SHT,
c) CMSX-4, 1100°C, 120MPa, 150h, d) CMSX-4, 1100°C, 117MPa, 392h

Fig1a is from an alloy, which contains no rhenium (CMSX-6), fig. 1b from the alloy with the highest rhenium content (CMSX-10). It is obvious that the size and concentration of pores is much higher in the rhenium containing alloy.
Fig. 1c shows an alloy with medium rhenium content (CMSX-4) after 150h creep at 1100°C, 120MPa and and fig. 1d the same alloy after 392h at 1100°C, 117MPa. At the shorter creep time we find round and square pores as well, but at the longer creep time square pores clearly dominate.

Discussion

The results are in good agreement with our previous investigations [1], where we measured the change of porosity either macroscopically via the density or microscopically from scanning microscope images of cross sections.
The increase of porosity with rising rhenium content results from the stronger segregation of this refractory element. Therefore a stronger homogenisation is necessary during which the pores are formed.
The different shape of the pores also fits with our expectation and is explained by the fact, that the round heat treatment pores form at much higher temperatures than the creep pores. Therefore the anisotropy of the interfacial enery should be lower.
The quantitative evaluation of the tomography images and the threedimensional visualisation is still under work. The reason is, that we have neither experience nor equipment for handling such hugh data sets. This problem is expected to be solved by cooperation with a tomografy group at the federal material institute in Berlin BAM (Dr. Goebbels) and a new computer with 2 Gb RAM.

Literature

1. Investigation of porosity in single-crystal Nickel-base superalloys, A. Epishin, T. Link, U. Brückner and P. D. Portella, proceedings of the 7th Liege conference, Materials for advanced power engineering, 2002.