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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **Evolution of pore morphology during shrinkage under hot isostatic pressing and growth by interdiffusion via Kirkendall mechanism in Ni-base alloys**	**Experiment number**: ME 1513
Beamline: ID19	**Date of experiment**: from: 24.10.2019 to: 26.10.2019	**Date of report**: 4/16/2019
Shifts: 9	**Local contact(s)**: Elodie Boller	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Bettina Camin, TU Berlin, Metallic Materials, Sekr. BH18, 10587 Berlin, Germany

*Alexander Epishin, TU Berlin, Metallic Materials, Sekr. BH18, 10587 Berlin, Germany

*Jonas Schmidt, TU Berlin, Metallic Materials, Sekr. BH18, 10587 Berlin, Germany

Walter Reimers, TU Berlin, Metallic Materials, Sekr. BH18, 10587 Berlin, Germany

Report:

Safety of aircraft flights depends greatly on the reliability of aircraft engines. Critical components of such engines are the high-pressure turbine blades in the hot section. To improve the strength of such blades they are solidified as single-crystals of nickel-base superalloys. However this technology cannot exclude microporosity, a dangerous microstructural defect, which forms during solidification and reduces the fatigue life of single crystal superalloys in a dramatic way. The reason is, that under cyclic loading typical for service conditions of turbine blades, rupture is initiated by crack nucleation at such micropores. Microporosity in single-crystal superalloys can be removed by Hot Isostatic Pressing (HIP) but the HIP-parameters like temperatur T, pressure p, and duration t have to be optimised to reach a full pore healing and minimize process costs. Opposite to this process is pore growth by interdiffusion via Kirkendall mechanism between the turbine blade and bond coat. In a first step the pore evolution during HIP was predicted by modelling. In a second step experiments were carried out to verify the modelling results and clarify the physical processes standing behind. The evolution of shape, size and distribution of pores in Ni-base superalloys CMSX-4 and CMSX-10, as well as in the diffusion couple CMSX-10/ Ni were investigated. These materials were investigated in different conditions -like as cast, heat treated, HIPed. Small samples with a diameter of d =1mm and a length l ≈10mm were cut by spark erosion for synchrotron tomography.

Four questions were addressed in this investigation:

1. The porosity evolution in superalloy CMSX-4 during HIP.
2. The kinetics of H(omogenisation)-porosity growth in superalloy CMSX-10 during homogenization heat treatment.
3. The growth and evolution of Kirkendall pores in diffusion couple CMSX-10/Ni during high temperature annealing.
4. The porosity growth in superalloy CMSX-4 during high temperature creep.

Overall around 210 measurements were carried out with a monochromatic beam at an energy of 70keV and a flux of 200mA (7/8 multibunch mode). A tomography sCMOS-camera PCO EDGE 4.2 featuring a high resolution of 2048 x 2048 pixel was used. During the rotation of the sample station about 360° overall 5000 radiographs were taken with an exposure time of 50ms each resulting in a total measuring time of appr. 5 min for one tomogram. The 360°-rotation of the sample leads finally to a total field of view of 3700 x 3700 x 2048 pixel imaging a frame of 1265 x 700 µm². From this it results that the resolution is appr. 0,34µm/ voxel. The samples were measured in absorption contrast and phase contrast tomography.
To clarify the first question samples in as-cast, heat treated, and HIPed conditions were analysed. Here HIP-temperature was constant, 1288°C, but other HIP-parameters, namely pressure *p* and time *t*, were varied. Also the effect of the initial condition (as-cast or heat treatment) -state before HIPing- was of particular interest. Fig. 1 shows the porosity and dendritic structure of the as-cast sample in a phase contrast image. As an example a selected void (yellow arrow) visible in the dendritic structure is shown in different views (x-y-, y-z-, x-z-planes, and 3D-volume). In the heat treated state (Fig. 2, phase contrast image) the dendritic structure is already invisible because in this condition material is chemically much more homogenious. However, the chemical homogenisation results in additional porosity, called H(omogenisation)-porosity. Finally it could be shown that the porosity caused by casting and heat treatment is reduced continuously by HIP until no more porosity could be detected.

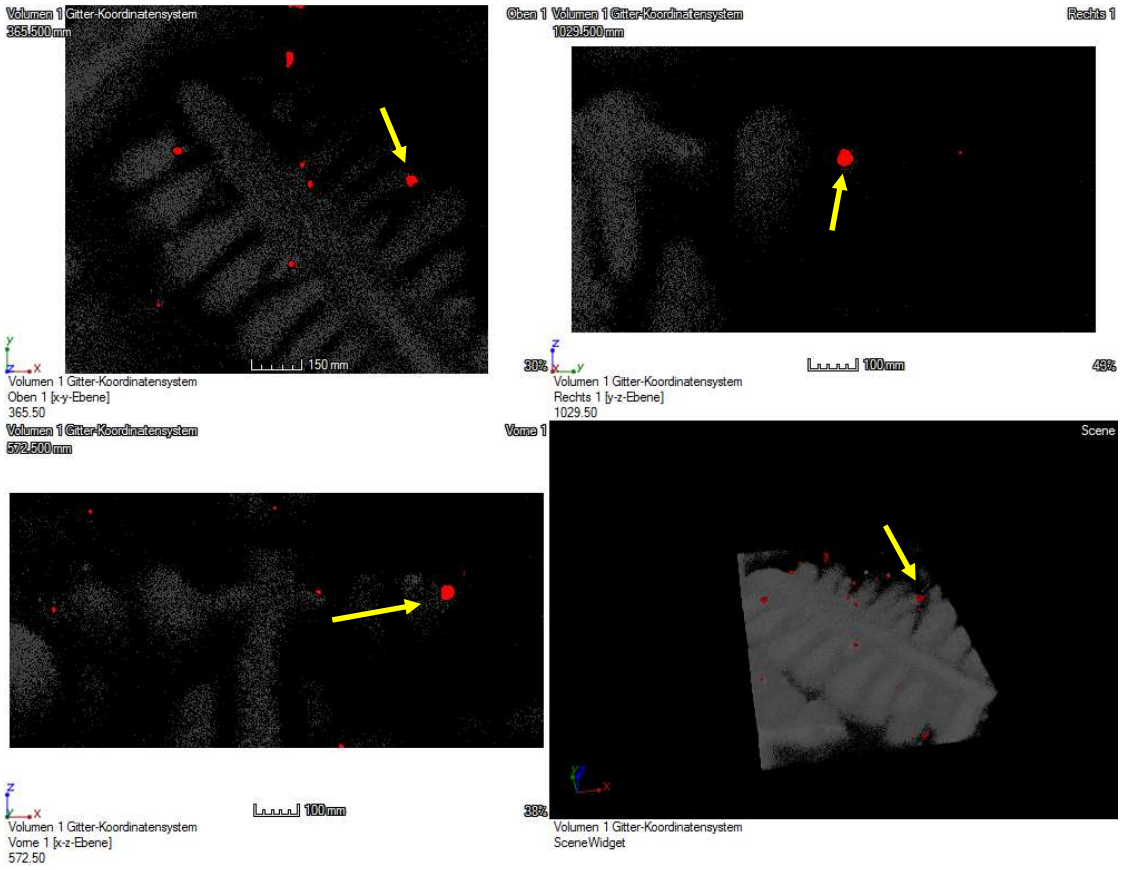


Fig. 1 *CMSX-4, initial as-cast state, different views of a selected void in the dendritic structure*

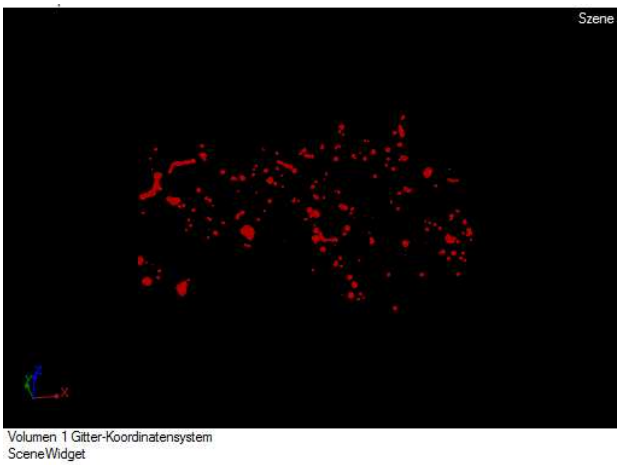


Fig. 2 *CMSX-4 after heat treatment, the porosity increases, the dendritic structure is not visible*

2nd question: For analysing the growth of H-porosity in CMSX-10 as-cast samples were homogenized at a temperature of 1350°C for different times between 2h and 128h at maximum. After a period of 4 hours the samples exhibit a weak dendritic structure and porosity (Fig. 3). With increasing time the dendritic structure becomes invisible due to chemical homogenisation but the porosity grows. This porosity is localized in the interdendritic regions and therefore arranged in form of rows between the dendritic arms (Fig. 4). The time depending correlation between homogenisation and porosity growth has to be analysed in further investigations.

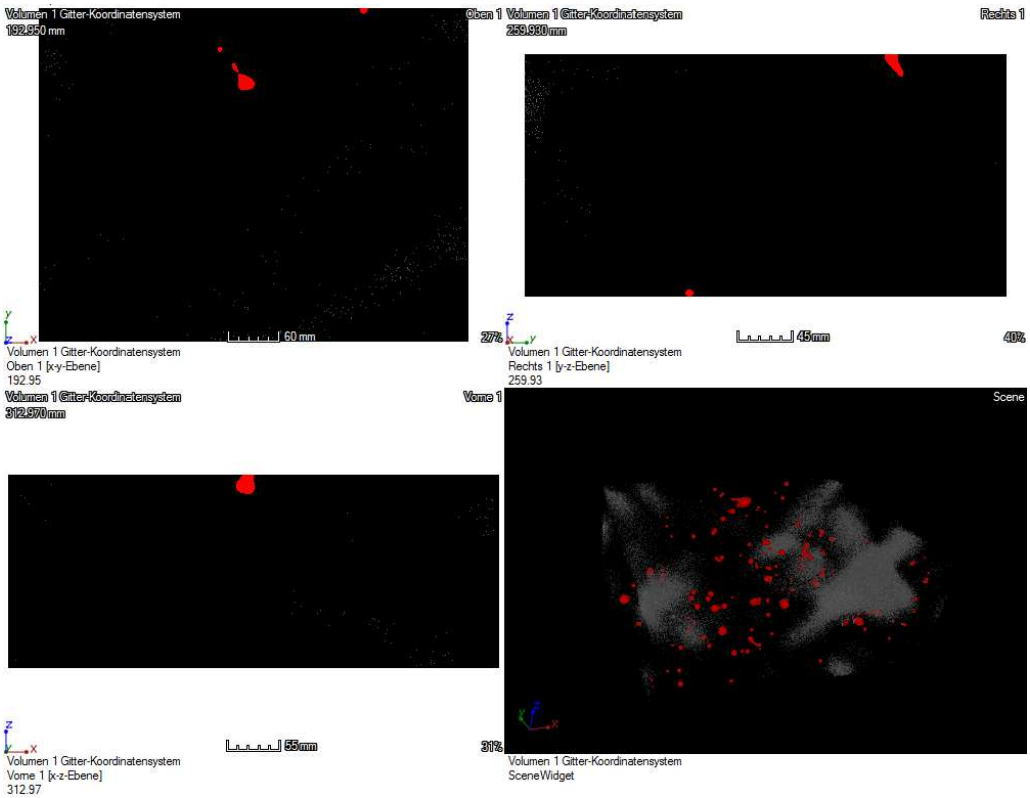
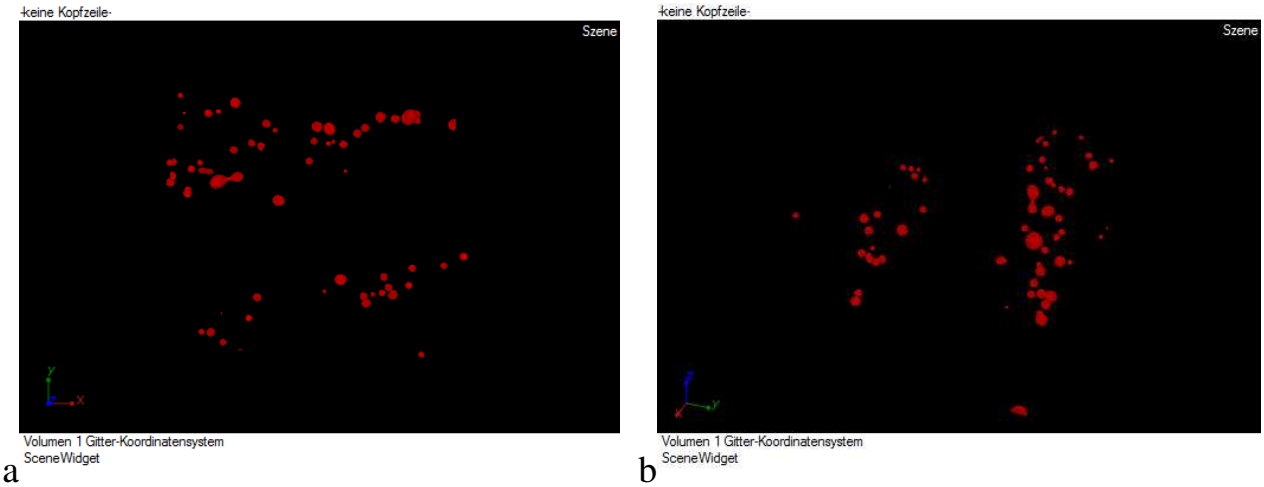


Fig. 3 *CMSX-10, as-cast and homogenized at 1350°C for 4h, weakly visible dendritic structure*



a b

Fig. 4 *CMSX-10, as-cast and homogenized at 1350°C for 128h, voids arranged in rows (a+b: different views)*

To answer the third question diffusion couples of CMSX-10 and Ni were investigated. The samples were annealed for 48h hours at three different temperatures: 1150°C, 1200°C, and 1250°C. It can be seen that in the diffusion zone (interface area) of the sample annealed at 1250°C for 48 hours the Kirkendall porosity (marked with a yellow brackett) exhibits a clear gradient (Fig. 5). This is caused to fast Al diffusion. Detailed analysis have to clarify the influence of temperature *T* and time *t* on the diffusion processes.

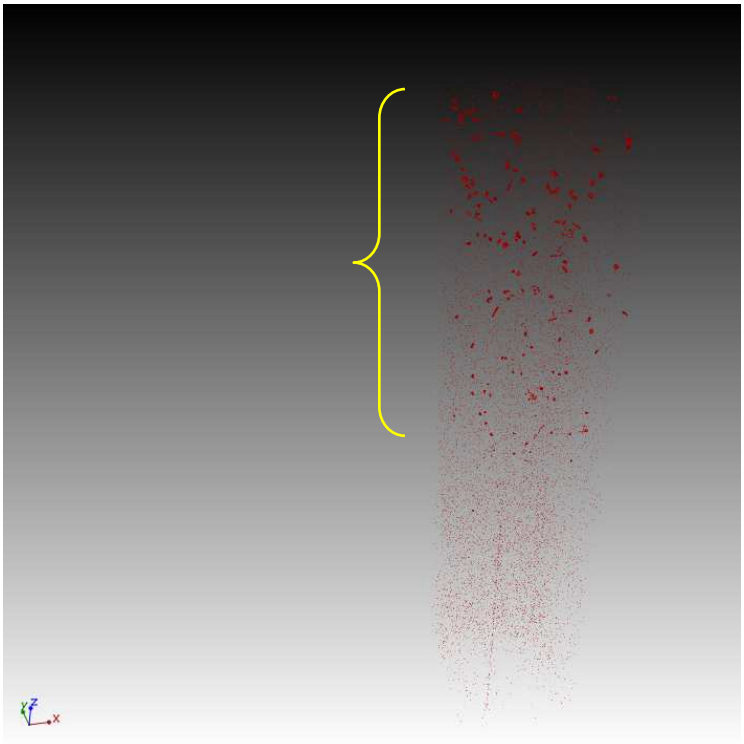


Fig. 5 *Porosity gradient in a diffusion couple CMSX-10/ Ni*

To investigate the evolution of porosity during creep (4th question), interrupted creep tests were carried out at different temperatures T and loads σ. Table 1 shows the creep conditions.

Table 1 *creep conditions*

temperature T [°C]	load σ [MPa]	time t_{creep} [h]
1050	90	1812
1100	90	100
		595
		663
		1200
		1800
	105	100
		106
		301
		334
		600
		837
1150	90	426

Figures 6 and 7 show the porosity after interrupted creep tests at a temperature of T=1100°C, load σ=105MPa after creep times of t_1=100h (Fig. 6) and t_2=600h (Fig. 7) choosing the same analysed volume size. The tomograms document that porosity is increasing with increasing time. The detailed analysis of the correlation between the porosity growth and the creep kinetics is under way and will help to understand the deformation mechanisms.

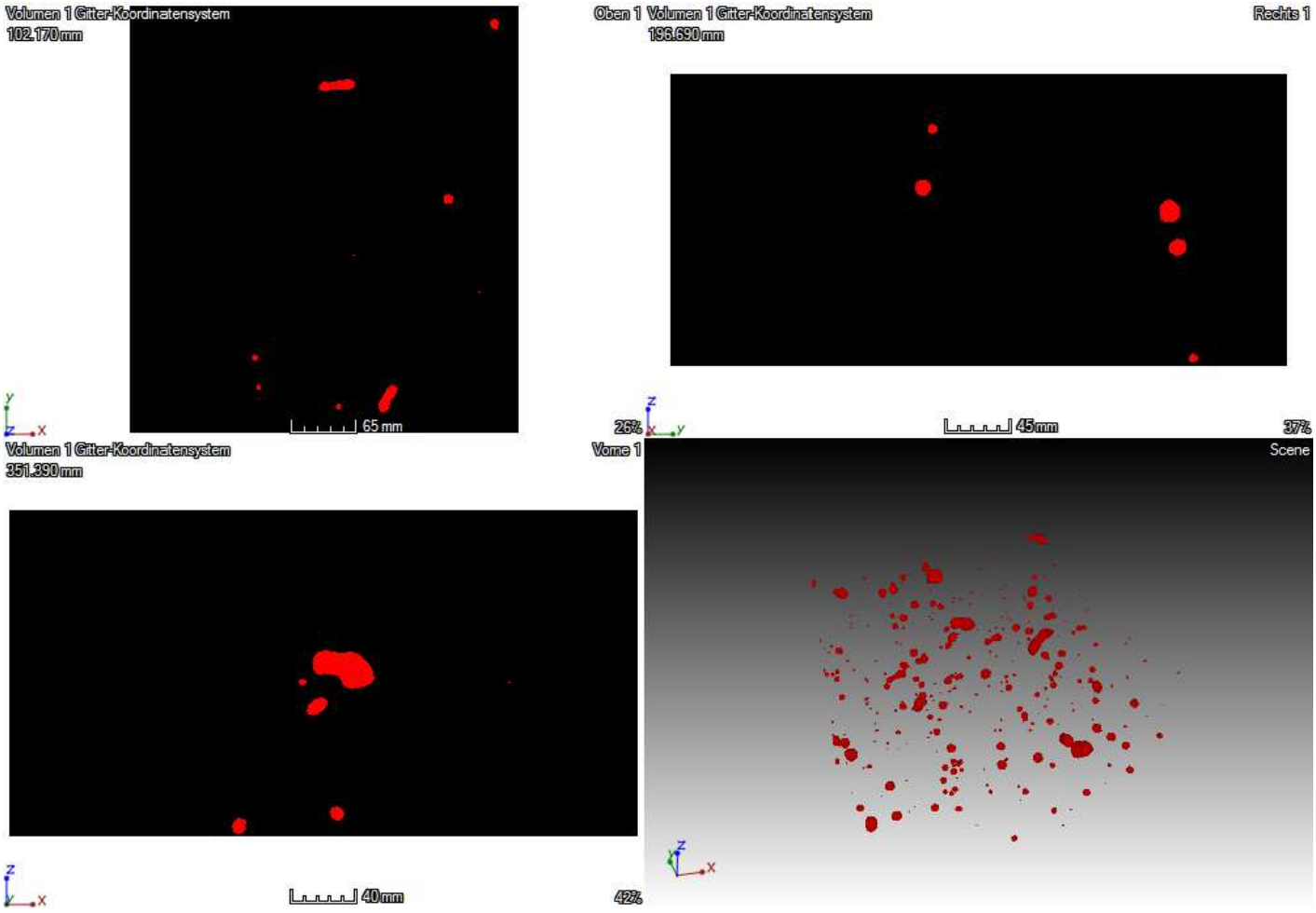


Fig. 6 *Porosity in deformed CMSX-4, creep condition: T=1100°C, σ=105MPa, t_1=100h*

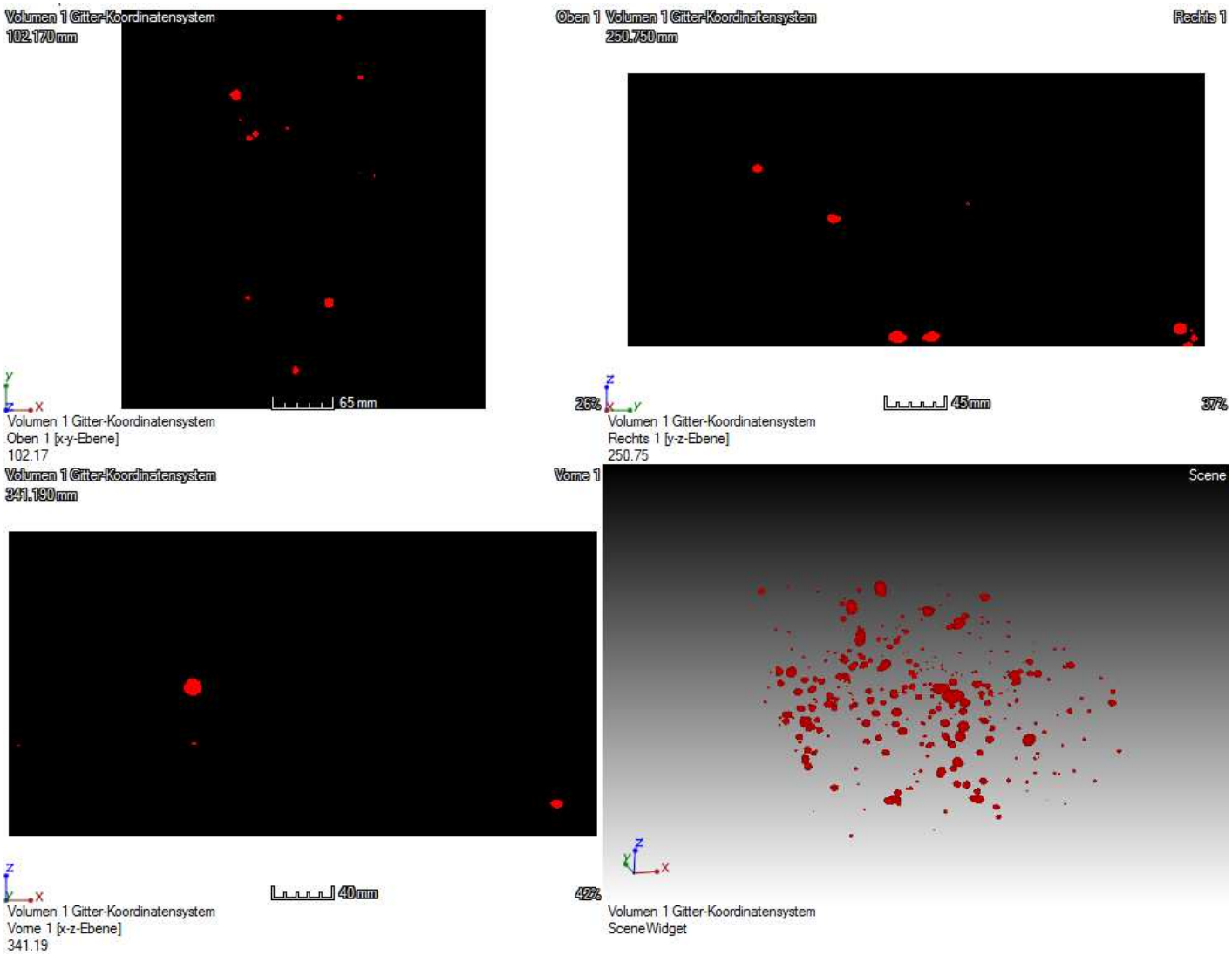


Fig. 7 *Porosity in deformed CMSX-4, creep condition: T=1100°C, σ=105MPa, t_2=600h*