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Grain boundary sliding in copper and its relation to cavity formation during creep

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ABSTRACT

The nucleation of creep cavities, which control the creep ductility is assumed to take place by grain boundary sliding. To determine the grain boundary sliding rate at longer testing times than previously available in the literature, two creep tests have been performed at constant loading rate at 125 °C for oxygen free copper with phosphorus (Cu-OFP). The tests were interrupted after certain strains and the amount of grain boundary sliding (GBS) was measured on flat polished surfaces. The observed amount of GBS per unit strain was 20 to 65 μm. This is of the same order of magnitude as for published tensile tests (Pettersson, 150 and 200 °C) and short time creep tests (Ayensu and Langdon, 400–600 °C). The amount of GBS was modelled based on previously performed FEM investigations. For conditions corresponding to the experiments a value of 52 μm was obtained.

A model by Lim for cavity nucleation at junctions between cell and grain boundaries has been adapted to oxygen free pure copper Cu-OF and Cu-OFP. The results show that the gain in free energy at cavity nucleation is much larger for Cu-OF than for Cu-OFP implying that Cu-OF is much more prone to cavity formation. The modelled difference in free energy gain is sufficient to quantitatively explain the much higher creep ductility in Cu-OFP than in Cu-OF.

1. Introduction

In Sweden the spent nuclear fuel is planned to be disposed underground by encapsulating in cast iron inserts enclosed by copper canisters [1]. The copper canisters will be made of phosphorus alloyed oxygen-free copper (Cu-OFP). The canisters will experience temperatures of up to 100 °C and an external hydrostatic pressure of about 15 MPa [2,3]. The hydrostatic pressure will give rise to creep deformation in the copper.

During creep, shear stresses will force the grains to slide against each other, so-called grain boundary sliding (GBS). GBS occurs along the grain boundaries as a bulk movement of two grains. GBS is one of the deformation mechanisms of materials at elevated temperature and low strain rate. GBS can give a significant contribution to the overall creep strain. The main mechanism of GBS is the combined motion of dislocations in and around the grain boundaries by glide and climb [4]. GBS takes place discontinuously with time and the amount of shear displacement is not uniform along the grain boundary.

An important effect of GBS is in the process of creep failure in terms of cavity initiation eventually leading to final rupture. Cavities are developed by GBS on the grain boundaries of pure copper specimens [5]. Langdon found that cavities are especially prevalent in pure copper at elevated temperatures and cavity nucleation is inhomogeneous [6]. However, there are many boundaries where no cavities are detected at all. It has been suggested that low angle and twin boundaries are creep cavitation resistant [7].

Observations and measurements on the creep strain attributable to GBS have frequently been performed on the specimen surface using scribe lines or micro-grid (see for example [8]) and more recently also using focused ion beam [9,10]. Using scribe lines on a polished and etched surface, the shear offset under application of stress σ can be observed and measured where the line crosses the grain boundary. Pettersson [11] used this technique to evaluate GBS in oxygen-free copper (Cu-OF) and Cu-OFP in tensile tests at temperatures up to 200 °C. The larger the plastic deformation, the larger was the mean sliding distance. The longest testing times in Pettersson's work for GBS measurements were 3 h. GBS measurements after creep tests for Cu-OF have also been performed by Ayensu and Langdon at 400–600 °C [12]. In this case the longest testing time was 5 h. There is obviously a need to measure grain boundary sliding during longer and more realistic creep times.

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Modelling of GBS with FEM has been performed in a number of papers, see for example [13,14]. However, there seems to be no attempt to compare these predictions to measurements on copper. It is well established that GBS gives rise to creep cavitation, but the exact mechanism has been controversial. High stresses are needed and they must be acting for a long time. High stresses can readily be formed for example around particles, but according to modelling results these stresses are quickly relaxed [15]. However, Lim formulated a model based on nucleation at cell and grain boundary junctions that avoided this problem [16]. His model is based on the formation of pile-ups of grain boundary dislocations that create persistent stresses on the junctions. The purposes of the present work are to (1) carry out creep tests for Cu-OFP and measure the amount of GBS; (2) model GBS based on published FEM investigations and (3) adapt and apply Lim's model to compute the free energy gain during cavity formation at cell boundaries in Cu-OF and Cu-OFP.

2. Materials and experiments

2.1. Material

The Swedish Nuclear Fuel and Waste Management Co (SKB) provided test material, which is phosphorus alloyed oxygen-free copper (Cu-OFP). The material was cast in vacuum and hot forged. The chemical composition is given in Table 1. The material contained mixed grains with a mean linear intercept grain size of 150 μm.

2.2. Round bar specimen with parallel planes

Round bar specimens were first manufactured with a diameter of 10 mm with a test length of 45.5 mm. Two parallel planes were then machined to facilitate observation and measurement of GBS, see Fig. 1. The parallel planes had a length of 18 mm, which was thus the gauge length of the specimens. The thickness of the parallel part was 6 mm.

Before creep testing, the planar surfaces were ground and polished to 0.25 μm, and finally etched in a solution containing 40 g CrO_3, 7.5 g HN_4Cl, 50 ml H_2SO_4, 50 ml HNO_3 and 1900 ml H_2O. Microstructure examination was then carried out on the etched planes using a Leica light optical macroscope (LOM), the Leica DM IRM, and a scanning electron microscope the Leo1530 upgraded to a Zeiss Supra 55 with a field emission-gun (FEG-SEM). On the etched planes, scratches were randomly added to be able to follow the movement at the grain boundaries. Scratches were made by sharp knife with an angle either perpendicular to or 45° off the stress axis.

2.3. Creep testing with constant loading rate

Creep testing was conducted on test rigs using a step motor actuating load through a gearbox and an angled gear. A load cell is incorporated in the load chain in this test setup and the step motor is controlled by feedback from the load cell. The load during the test was regulated within 1 N of target load, fulfilling the requirement of ASTM E139-2011 standard [17]. An in-house developed programme controlled the load increment at a given loading rate. In the present case the loading rate was constant. The furnace is of hot air type and temperature is controlled within ± 1 °C.

Initially it was considered to perform the creep tests at constant load in the usual way. However, this (or for tests at constant stress) would give an initial plastic strain in the copper of 10% or more. It was anticipated that it would make the measurements of GBS more difficult due to extensive lattice deformation. For this reason tests with a constant loading rate were chosen instead. In fact, the results of the present investigation showed that for larger strains, fewer GBS events could be recorded.

After testing, GBS on both specimens was observed and measured using light optical microscopy (LOM) and a field emission gun-scanning electron microscope (FEG-SEM). Silicon was applied on the planar surfaces to provide protection against oxidation. The silicon was removed before observation of GBS.

Table 1
Chemical composition of the investigated Cu-OFP in wt. ppm, except for Cu (wt%).

Cu	P	Ag	Al	As	Bi	Cd	Co	Cr	Fe	H
99.991	66	7.7	< 0.08	0.3	< 0.3	< 0.4	< 0.1	< 0.05	0.8	0.58
Mn	Ni	O	Pb	S	Sb	Se	Si	Sn	Te	Zn
< 0.1	1.3	3.1	< 0.7	3	< 1	< 0.7	< 0.2	0.7	< 2	< 0.1
Zr										
< 0.2										

3. Results

3.1. Creep testing

The creep testing results at 125 °C are given in Table 2. Two tests were performed. The test using specimen T238 with a constant loading rate of 12 N/h (238 MPa/h) was interrupted after 307 h. At interruption, the stress and the strain were 73.7 MPa and 3.36%, respectively. Since the loading rate was constant, the (engineering) stress linearly increased with time.

The strain as a function of time to 307 h is presented in Fig. 2. A non-linear relation between strain and time is observed. After commencement of test, a weak primary stage is observed. A minimum strain rate of about 1×10^{-4}/h is found at about 240 h (corresponding to a strain of 2.3% and a stress of 57 MPa, see Fig. 2). Then, the strain increases faster with time. A maximum strain rate of about 3×10^{-3}/h was recorded just before the interruption.

The test with the specimen T238 was then restarted from zero load at the same loading rate as before the interruption and continued until a total testing time of 502 h had been reached, see Fig. 2. At the 2nd interruption, the stress was 46.8 MPa and the accumulated strain 20.8%, see Fig. 3, where the stress as a function of strain is shown for the whole test. It is apparent that the strain increases faster after restart, see Fig. 2. This is also evident from the stress versus strain relation, Fig. 3, where the strain increases faster with increasing stress after restart. The strain rate at the 2nd interruption had doubled in comparison to that at the 1st interruption.

Strain and stress curves for the test with specimen T056 with a constant loading rate of 2.82 N/h are also presented in Figs. 2 and 3. The stress increase with time is 0.056 MPa/h. Also for this test a weak primary stage is found, Fig. 2. A minimum strain rate of about 2×10^{-5}/h is observed after 450 h (corresponding to a strain of about 1.6%, and a stress of about 25 MPa, see Fig. 2). A maximum strain rate of about 1×10^{-4}/h is recorded at interruption. The stress–strain relation is shown in Fig. 3.

Creep damage may have contributed to faster strain accumulation after restart. At first interruption for the specimen T238, no creep damage was observed. After the second interruption after 507 h and 20.8% strain, wedge cracks and intergranular cracks were found. No cracks were found in the tests with lower creep strain. Creep damage in the form of cavitation could be detected at later stages of the creep test [18,19]. In the present case, many cracks were found at 20% strain, indicating that intergranular and brittle rupture may occur. No creep damage was found in the