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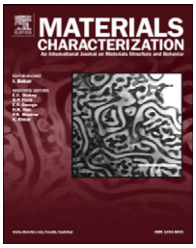

Cell structure in cold worked and creep deformed phosphorus alloyed copper

Rui Wu[a], Niklas Pettersson[a], Åsa Martinsson[a,c], Rolf Sandström[a,b,*]

[a]Swerea KIMAB, Box 7074, SE-164 07 Kista, Sweden
[b]Materials Science and Engineering, Royal Institute of Technology, SE-100 44 Stockholm, Sweden
[c]Now at Sandvik Materials Technology, Sandviken, Sweden

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ABSTRACT

Transmission electron microscopy (TEM) examinations on as-received, cold worked, as well as cold worked and creep tested phosphorus-alloyed oxygen-free copper (Cu-OFP) have been carried out to study the role of the cell structure. The cell size decreased linearly with increasing plastic deformation in tension. The flow stress in the tests could also be correlated to the cell size. The observed relation between the flow stress and the cell size was in excellent agreement with previously published results. The dense dislocation walls that appeared after cold work in tension is likely to be the main reason for the dramatic increase in creep strength. The dense dislocation walls act as barriers against dislocation motion and their presence also reduces the recovery rate due to an unbalanced dislocation content.

1. Introduction

In Sweden spent nuclear fuel is planned to be disposed of by encapsulating in cast iron inserts placed inside copper canisters [1]. The cast iron insert is the load bearing part of the waste package and the copper canister acts as corrosion barrier. Due to an external hydrostatic pressure, the copper canister will be exposed to creep deformation [2,3].

The cylindrical copper canister is about 1 m in diameter, 5 m long, and has a wall thickness of 50 mm. It will be made out of phosphorus alloyed oxygen-free copper (Cu-OFP). After the hot working process which is either in the form of extrusion, pierce-and-draw or forging, the canister is in a soft condition. During the subsequent handling, the canister may be subject to local cold working. For example, an incorrect application of a tool might introduce indenting.

It is well known that dislocation movement during plastic deformation forms cells and subgrains in many alloys. Cells are built up of nearly dislocation-free regions which are, separated by loosely knit tangles of dislocations. On the other hand, subgrains are separated by boundaries of sets of parallel dislocations [4].

It is well established that the subgrain size d that is developed during creep is frequently inversely proportional to the creep stress σ [5]. Sometimes a stronger stress dependence is observed at higher stresses [6]. In stress dip tests where the creep stress is suddenly reduced while the microstructure is unchanged, it has been found that the creep rate is proportional to the cube of the subgrain size. A survey can be found in [6]. It thus seems that the control of the subgrain size would be a powerful way of improving the creep strength. However, the subgrain size is not stable, and as just pointed out, the subgrain

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* Corresponding author.
E-mail address: rsand@kth.se (R. Sandström).

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is generally controlled by the creep stress. To make use of an increased creep strength due to a fine subgrain size, the subgrain size must be stabilised. This is well established for 9–12% Cr-steels, where the carbides are stabilising the subgrain size [7].

Cold deformation reduces the cell and subgrain sizes [8]. It has been found in many investigations that prior cold working can increase the creep strength of fcc alloys. In particular, studies have been performed for austenitic stainless steels [9–12] and nickel-base alloys [13,14]. For prior cold working up to 10 to 20%, an improvement in creep strength has typically been found. A number of mechanisms have been proposed for this effect. The cold working raises the dislocation density. According to the recovery creep theory, the dislocation density should be reduced to the same stationary value as for material without cold working. Hence, to get an effect of the cold working the dislocation density must be stabilised at a higher level. It has been proposed that the subgrain boundaries [15] or the dislocations inside the subgrains [10] would provide this stabilisation. Such assumptions would explain why the effect of cold working is reduced at high temperatures where the recovery is a faster or at low stresses where the recovery has more time to operate. Another proposed mechanism is that particle precipitation on the dislocation takes place and in this way stabilises the dislocation network [12].

Many fcc materials including copper form substructure both at ambient and elevated temperatures. The role of the substructure concerning the influence on mechanical properties is still unclear in spite of the fact that it has been studied over a long period of time. In particular, it is urgent to perform studies where detailed creep investigations are combined with careful metallographic characterisation.

The purposes of the paper are

- to establish a defined and reproducible sample preparation process for TEM examination for cold worked Cu-OFP,
- to examine cold worked and creep tested samples in TEM,
- to correlate the cell size with the degree of cold working and test stress, and
- to explain how the creep properties are affected by cold working.

2. Materials and Experiments

2.1. Material

The Swedish Nuclear Fuel and Waste Management Co (SKB) provided test materials which were pure, oxygen-free phosphorus-doped copper (Cu-OFP). The materials were extracted from two forged lids and have the SKB internal identity TX104 and TX184, respectively. The chemical compositions are given in Table 1. Reference material was taken from the forged lid TX104, which was the as-received material. The reference material had a hardness of 32.2 (HV0.5) and an average grain size of 150 μm.

2.2. Cold Working and Creep Testing

The materials from the forged lids TX104 and TX184 were subjected to cold working in tension and in compression followed by creep testing. Creep testing results are illustrated in Figs. 1 and 2. With cold work of 12 and 24% in tension, the creep strength is significantly increased. On the other hand if the cold work is performed in compression little increase in creep strength is observed. In all cases the creep rupture elongation is reduced when cold work is introduced prior to the creep testing. Further details on the extraction of specimens and the results of the creep testing of the cold worked copper can be found in [16].

The specimens listed in Table 2 were used for transmission electron microscopy (TEM) investigations. Five specimens were studied. Specimen 1 was reference material without any additional cold working after forging. A typical start temperature for the forging of canister lids is 675 °C [17]. The finishing temperature that controls the substructure can be estimated to 450 °C considering the chosen start temperature [18]. During forging the material is exposed to large strains, which corresponds to a flow stress of about 160 MPa at 450 °C [19].

Specimens 3 and 4 were cold worked to 12 and 24% in tension, respectively, but not creep tested. Specimens 6 and 10 were first cold work and then creep tested. In addition to the difference in the amount of cold work, specimen 6 was cold worked in compression and specimen 10 in tension. Both specimens were creep tested in tension to rupture. For the specimen 6, compression direction in cold working and tension direction in creep were opposite. For the specimen 10, tension direction in cold working and in creep was the same. The creep test results for the specimens 6 and 10 are given in Table 2.

2.3. Thin Foil Sample Preparation

TEM investigations were performed on thin foils taken from the reference material (specimen 1), from the cross section of the tensile specimens (specimens 3 and 4) and from the creep specimens (specimens 6 and 10). For the specimens 3 and 4, the foils were sectioned from the uniformly deformed part within the gauge length of the tensile specimens. For the

Table 1 – Chemical compositions of the copper material in units of wt ppm.

Component	Test series	Pb	Bi	As	Sb	Sn	Zn	Mn	Cr	Co
TX184	12% in compression	<1	<1	<1	1	<0.5	<1	<0.5	<1	<1
TX104	Lid ref, 12% and 24% in tension	<1	<1	<1	1	<0.5	<1	<0.5	<1	<1
Component	**Test series**	**Cd**	**Fe**	**Ni**	**Ag**	**Se**	**Te**	**S**	**P**	**O**
TX184	12% in compression	<1	1	2	11	<1	<1	6	41–67	1
TX104	Lid ref, 12% and 24% in tension	<1	2	2	13	<1	<1	5	45–60	1–2