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An updated review of the creep ductility of copper including the effect of phosphorus

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SSM:s perspektiv

Bakgrund

Strålsäkerhetsmyndigheten (SSM) granskar Svensk Kärnbränslehantering AB:s (SKB) ansökningar enligt lagen (1984:3) om kärnteknisk verksamhet om uppförande, innehav och drift av ett slutförvar för använt kärnbränsle och av en inkapslingsanläggning. Som en del i granskningen ger SSM konsulter uppdrag för att inhämta information i avgränsade frågor. I SSM:s Technical note-serie rapporteras resultaten från dessa konsultuppdrag.

Projektets syfte

Uppdraget är en del i SSM:s granskning av SKB:s ansökan om slutförvaring av använt kärnbränsle. Uppdraget avser granskning av SKB föreslagna krypbrottmekanismer för det kopparmaterial som planeras användas som korrosionsbarriär i kapseln. Detta arbete är en fortsättning på ett tidigare arbete 2010-2011 som redovisats i SSM Technical Note 2012:13.

Författarens sammanfattning

I ett tidigare arbete, se SSM Technical Note 2012:13, drogs slutsatsen att SKB har presenterat otillräckliga bevis för att motivera sin ståndpunkt att fosforlegerad syrefri (OFP) koppar har en tillräcklig krypduktilitet under långtidsförvaring. Denna slutsats baserades på en studie om korngränsglidning i både OF och OFP koppar material vilken visade att grunden för SKB: s teori om krypsprödhet, som är nödvändig för extrapolering till lagringsförhållanden, var felaktig. Nyligen har SKB försökt att ogiltigförklara resultatet av studien om korngränsglidning. Det arbete som redovisas i denna rapport visar dock att resultaten för syrefri (OF) koppar från studien om korngränsglidning är förenliga med SKB:s teori (vilken har kalibrerats mot SKB:s resultat från OF koppar). SKB har enligt författaren i otillräcklig omfattning lyckats med att förklara varför OFP koppar med samma mängd korngränsglidning som OF koppar förblir segt under förhållanden där OF koppar beter sig på ett krypsprött sätt. Den tidigare dragna slutsatsen kvarstår därmed.

Projektinformation

Kontaktperson på SSM: Peter Ekström
Diarienummer avrop: SSM2014-1911
Aktivitetsnummer: 3030012-4116

SSM perspective

Background

The Swedish Radiation Safety Authority (SSM) reviews the Swedish Nuclear Fuel Company's (SKB) applications under the Act on Nuclear Activities (SFS 1984:3) for the construction and operation of a repository for spent nuclear fuel and for an encapsulation facility. As part of the review, SSM commissions consultants to carry out work in order to obtain information on specific issues. The results from the consultants' tasks are reported in SSM's Technical Note series.

Objective

This project is part of SSM:s review of SKB:s license application for final disposal of spent nuclear fuel. The assignment concerns review of creep mechanisms for copper material used for corrosion barrier in canisters for final disposal of nuclear fuel in Sweden. This work is a continuation of an earlier work in 2010-2011 reported in SSM Technical Note 2012: 13.

Summary by the author

In a previous report, SSM Technical Note 2012:13, it was concluded that SKB has presented insufficient evidence to justify their position that the phosphorus alloyed oxygen free (OFP) copper has an adequate creep ductility during long term storage. This conclusion was based on a study of grain boundary sliding in both OF and OFP copper, which showed that the basis for the SKB theory on creep brittleness, necessary for extrapolation to storage conditions, was wrong. Recently SKB has attempted to invalidate the results of the grain boundary sliding study but in the present report it has been shown that the results for oxygen free (OF) copper are consistent with SKB's theory (which is calibrated against their results for OF copper). In the author's view, this implies that SKB has presented insufficient explanation for why OFP material, despite having the same amount of grain boundary sliding as OF copper, remains ductile under conditions when OF behaves in a brittle creep manner. Therefore the previous conclusion stands.

Project information

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This report was commissioned by the Swedish Radiation Safety Authority (SSM). The conclusions and viewpoints presented in the report are those of the author(s) and do not necessarily coincide with those of SSM.

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1. Background

The copper used as the corrosion barrier of the canisters which will be used for long term storage of nuclear waste in Sweden is a pure copper with an addition of 30-100 ppm of phosphorus (OFP copper). The purpose of the addition of phosphorus is to ensure that the copper will have adequate creep ductility[1] under the conditions of long term storage. Early experiments with copper without the addition of P (OF copper) showed that this material had a very low creep ductility when tested in the temperature range 180 - 215 ˚C [1]. Since there was no guarantee that this would not also occur at the lower temperatures of waste storage it was necessary to find a copper material with better creep ductility. It turned out that copper with an addition of about 50 ppm P appears to be such a material [1]. After an extensive testing programme which seemingly confirmed the high creep ductility of P doped copper it has been chosen by the Swedish Nuclear Fuel and Waste Management Company (SKB) as the material for the corrosion barrier of their planned waste canisters.

In a previous review Matsafe AB cast some doubt on the SKB position that copper with a phosphorus addition has an adequate ductility for the waste storage application [2]. All SKB experiments have resulted in ductile failure and therefore SKB has concluded that the OFP copper has an adequate ductility for storage. However, Matsafe AB has pointed out that storage conditions are so different from the SKB experimental conditions that it is conceivable that the low ductility failure mechanism might be active under waste storage without ever being observed under SKB experimental conditions. Such considerations probably gave SKB the incentive to develop a theory for the effect of phosphorus on the creep ductility of copper since any credible statement on copper ductility under waste storage conditions must rely on extrapolations from experimental conditions [3, 4]. The theory was based on the premise that phosphorus retards the grain boundary sliding in copper. Since it is the grain boundary sliding which nucleates the grain boundary cavities which cause the low creep ductility the elimination of grain boundary sliding also eliminates the low creep ductility.

However, an experimental study conducted by Matsafe AB showed that there was about the same amount of grain boundary sliding in OF as in OFP copper [5], thus invalidating the SKB creep ductility theory. In recent reports SKB defends the validity of the theory based on the argument that the experiments carried out by Matsafe AB were conducted for short time periods and that at the longer times of the SKB experiments and at time periods relevant to waste storage there could still be the assumed difference in grain boundary sliding between OF and OFP copper.

2. Objective of the review.

The purpose of the present review is to make a fair assessment of the SKB position with regard to the differences in grain boundary sliding between OF and OFP copper and the effect of that difference.

[1] Creep ductility can be defined as strain to final fracture in a creep test.

3. SKB's position.

SKB's position on the creep ductility issue can be found in four recent technical reports:

1. Design, production and initial state of the canister, TR-10-14, December 2010.

2. Design analysis report for the canister, TR-10-28, April 2010.

3. Fuel and canister process report, TR-10-46, December 2010.

4. The role of phosphorus for the mechanical properties of copper, SKBdoc 1417069 v.1 by R. Sandström.

In the summary of 1 creep ductility is listed as one of the most important properties of the canister in order to perform its barrier function. In the report it is stated that in order to obtain the required creep ductility it is necessary to add 30 -100 ppm of P to the material and to keep the sulphur content below 12 ppm. The required creep ductility is listed as > 15 %. It is not exactly clear how this number has been determined, it should probably come out as a result of the Design analysis report but it is nowhere to be found in that report. The process report contains many interesting discussions of failure mechanisms for both insert and shell but no discussion of low creep ductility. SKB's experimental creep testing programme is largely summarized in a report by Andersson-Östling and Sandström [6]. To summarize the initial SKB position: The copper used for the canister needs to have a creep ductility > 15 % and that requirement is fulfilled by the highly pure copper (>99.99 %) with 30─100 ppm P and less than 12 ppm S. This position has subsequently been supplemented by Report No. 4 [7] which deals with most theoretical aspects of the effects of phosphorus. In particular it presents a short argument for why the grain boundary sliding results, which showed that phosphorus has no effect, are irrelevant.

4. Evaluation and analysis of SKB's position.

4.1. Presentation of the problem

The problem with SKB's position that the highly pure copper with a P addition, OFP copper, has been shown to have an adequate ductility for long term nuclear waste storage has been discussed in a previous SKI Report [8]. The basis for the discussion is a plot of results from the original report [1] where copper without the P addition, OF copper, was found to have a very low ductility compared to OFP copper. The plot shown as Figure 1 shows the time to failure as a function of stress.

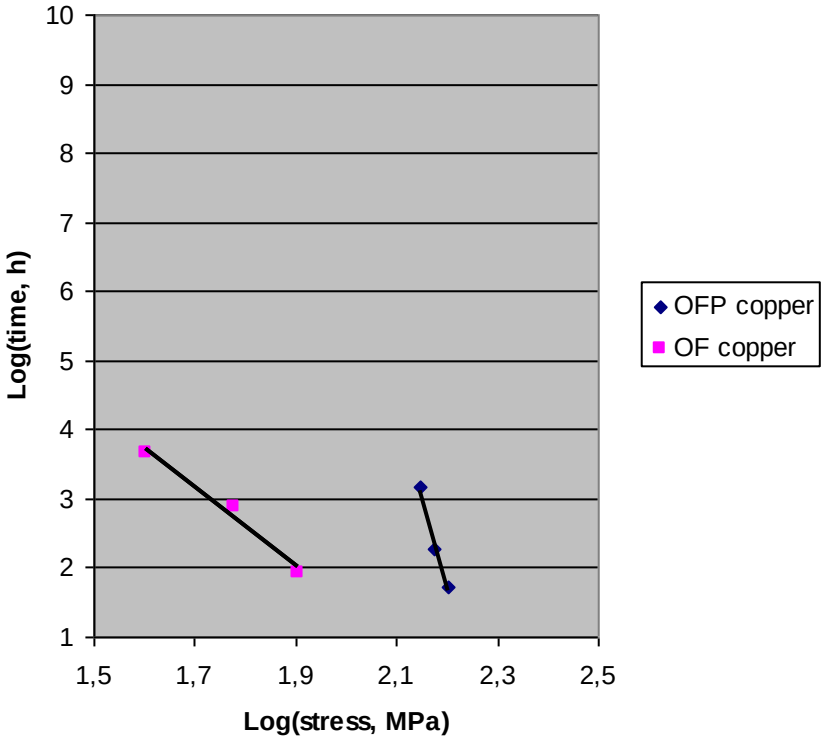

Figure 1. *Time to failure in creep tests of OF and OFP copper at 215 ˚C.*

It is clear that the stress dependence of the time to failure is different in the two materials. This is not very surprising since the processes leading to failure are different for the two materials. In the OF material, failure starts by the nucleation of small cavities in the grain boundaries. These cavities grow and when a certain fraction of the boundary is covered with cavities the grains separate from each other. When a sufficient fraction of the grains have separated the specimen fails by creep brittle failure. Observations in the SKB experiments show that the failure strains can be < 1 % [1].

In the OFP material the specimens fail by thinning down as a result of the creep strain until no load bearing area remains. Initially the specimen is thinned down uniformly along the specimen as the deformation increases. However, at some stage a neck of localized higher deformation than elsewhere on the specimen will form. In the type of creep experiment performed by SKB, creep under constant load, it may be shown theoretically that the neck starts to form at the inflection point of the creep curve which is also the point of minimum creep rate during a constant load creep experiment. It should be noted that many of the creep experiment performed by SKB are such that the thinning down of the specimen has a notable effect on the applied stress. In many cases the minimum creep rate occurs after about 10-20 % strain and the true stress is therefore 10-20 % higher than the initially applied stress.

The slopes of the stress dependencies in Figure 1 reflect how the two different failure processes depend on stress. In the case of OFP it will be the negative of the stress exponent for the creep rate while for OF it will be a so far unknown quantity. It is probable that the creep brittle process depends on tensile stress while the deformation induced failure process in OFP depends on shear stress.

Despite attempts to explain the effect of P on creep brittleness [1, 3] it is probably fair to say that the cause of the effect is not fully understood. In the absence of any understanding we can do the following thought experiment. Let us assume that the effect of P is to decelerate the creep brittle process by a factor of 10000. This gives one of the thin lines in Figure 2. The other thin line is an extrapolation of the OFP results.

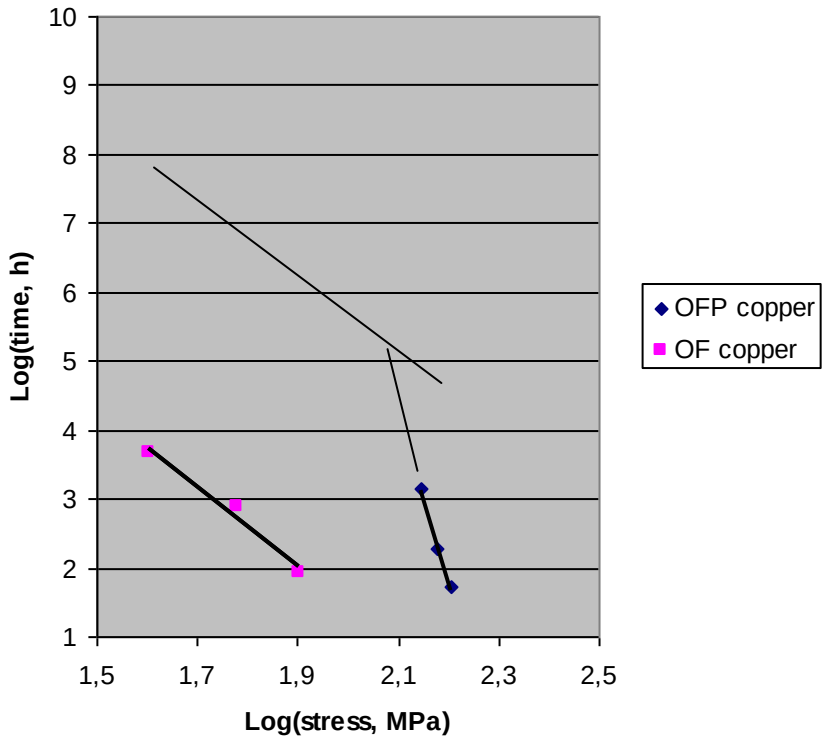

Figure 2. *Time to failure in creep tests of OF and OFP copper at 215 ˚C. The line parallel to the OF results represent a hypothetical situation where P decelerates the creep brittle process by a factor of 10000.*

The two thin lines cross at slightly less than 100 MPa giving a ductile failure time of just over 100000 h or about 12 years. Since none of the SKB creep tests have gone on for such a long time it is clear that the large volume of creep experiments performed by SKB would have told us nothing of a possible creep brittleness of OFP, if such a creep brittleness had the properties represented by the thin line in the Figure 2. But it would still be a creep brittleness of concern for long-term storage.

The discussion around Figure 2 was first presented in 2006 [8]. A few months later SKB issued a theoretical analysis of the creep brittleness of OF and OFP copper by Sandström and Wu [3]. One of the basic premises for the analysis was that part of the phosphorus content of OFP is located in the grain boundaries. The presence of P in the grain boundaries impedes grain boundary sliding which was postulated to cause nucleation of cavities in the grain boundaries. Thus there will be much less nucleation of cavities in OFP and therefore no creep brittleness. Another interesting result of the theory was that at low temperature there would be no creep brittleness in neither of the two materials. It should be noted that SKB does not rely on this theory in their license application. The statements on creep ductility are based solely on results from creep experiments run for periods which are 5 to 6 orders of magnitude shorter than the intended storage time.

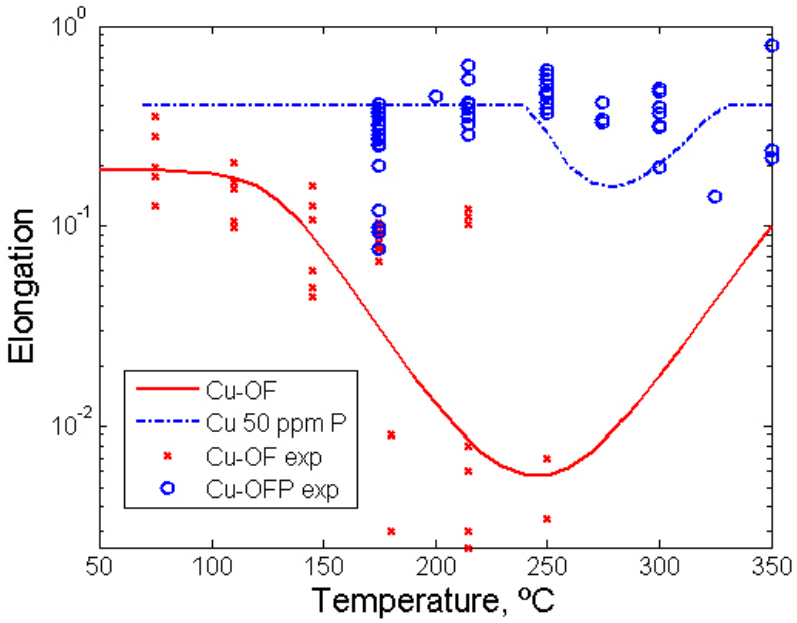

Figure 3. *Theoretical evaluation of the creep brittleness of OF and OFP copper by Sandström and Wu [3]. The curves show predictions by the theory and the plotted points are experimental results for a rupture time of 10000 h.*

As can be seen in Figure 3 both OF and OFP has an adequate ductility at low temperature. A credible theory of the type presented by Sandström and Wu would alleviate the concerns raised in connection with Figure 2.

4.2. The grain boundary sliding study

However, there is a problem with the Sandström-Wu theory. About ten years earlier the present writer hypothesized that P in the grain boundaries would impede grain boundary sliding and thus explain why OFP was more creep deformation resistant than OF. The idea was tested in a small student project where observations on grain boundary sliding was compared between OF and OFP after tensile testing at 100 and 200 ˚C to low strain. The results were too few to allow any certain conclusions but the general impression was that there was no difference between the materials with regard to grain boundary sliding. These observations justified a more extensive investigation of grain boundary sliding in OF and OFP copper.

This investigation was carried out with financing from SSM and the results were published in 2010 [5]. The main result of the investigation was that there was no significant difference in the grain boundary sliding in OF and OFP copper when the materials had been deformed to the same strain. This was true both at 100, 150 and 200 ˚C. Thus the basic premise of the Sandstöm-Wu theory has been shown to be incorrect.

A few other interesting observations were done in the project. As long as no cracking has occurred there is no difference at all in the mechanical properties of OF and OFP copper. But as soon as cracking started, the OF material deformed at a

lower stress than the OFP material. This observation indicates that much, if not all, of the difference in creep strength between the OF and OFP materials may be a result of creep cracking in the former.

4.3. Objections to the grain boundary sliding study

In a recent paper, Sandström and Wu try to invalidate the results of the grain boundary sliding study [4]. A main objection is that the testing times were too short to be relevant for SKB creep results. This objection will be discussed in the first subsection of the present section. The second subsection will be devoted to a discussion on how much P one would expect in a copper grain boundary based on diffusion calculations. In the final subsection it will be shown that the creep fracture results of the OF materials used in the grain boundary sliding study are consistent with the creep fracture theory of Sandström-Wu [3] which indicates that it is a manifestation of the same phenomenon as that observed in longer term creep tests which have been used to calibrate the Sandström-Wu theory.

4.3.1. How long does it take to reach the equilibrium P concentration in a grain boundary

In the grain boundary sliding study [5] testing times were at most about 5 hours while SKB creep testing times were of the order of 10000 h [4]. It might be more appropriate to compare strain rates. If one assumes that SKB specimens of OFP which failed in a ductile manner had failure strains of 40 % then the specimens of the grain boundary sliding study had strain rates 200 to 40000 times higher than the SKB creep test specimens. However, Sandström and Wu speculate that it is the time which is important and state that 5 hours is too short a time for phosphorus to diffuse into the grain boundaries and then lock their movement. As will be shown in the following paragraphs this is at least partly a valid point.

Sandström and Wu use an interaction energy $-\Delta E = 2.2 \times 10^{-20}$ J between P atoms and grain boundaries. Thus if the concentration of P in the matrix is x_P then the concentration in the grain boundaries x_{PGB} is given by

$$x_{PGB} = x_P \exp\left(-\frac{\Delta E}{kT}\right) \qquad (1)$$

The factor after x_P shows how many times higher the concentration is in the grain boundary than in the matrix under equilibrium conditions. The magnitude of the factor at different temperatures is shown in table 1 below.

If it is assumed that the thickness of the grain boundary is about one atom plane (≈ 0.25 nm) the factor shows how many atom planes need to give up their P content in order to provide the segregant to the grain boundary. Since a grain with a 100 µm grain size contains 400000 atom planes it is immediately realized that the grain boundary segregation has no significant impact on the matrix content of P.

Table 1. Calculated factor, x_P, displays at different temperatures how many times higher the phosphorus concentration is in the grain boundary compared to the matrix concentration, under equilibrium conditions.

Temp, ˚C	factor
0	343
50	139,2
100	71,8
150	43,3
200	29,1
250	21,1
300	16,2
350	12,9
400	10,7

It is likely that the grain boundary contains at least about 10 - 15 times the matrix content since the grain boundaries sweep up matrix P when they move during recrystallization and grain growth at around 300 - 400 ˚C. However the question is how fast the equilibrium segregation is reached at lower temperature. This is a one-dimensional diffusion problem which can be solved numerically with a finite-difference calculation. It is not possible to reach equilibrium in a finite time so therefore it was calculated how long time it would take to reach 60 % and 90 % of the equilibrium grain boundary concentration at different temperatures. For the diffusion of P the following expression was used [9]:

$$D = 4.4 \cdot 10^{-7} \exp\left(-\frac{138000}{RT}\right) \text{ m}^2/\text{s} \qquad (2)$$

The result is summarized in Table 2.

Table 2. Time to reach 60% and 90% of equilibrium phosphurus concentration in a grain boundary

Temperature ˚C	Time to reach 60% concentration	Time to reach 90% concentration
50	563000 years	15400000 years
100	160 years	3950 years
150	102 days	7 years
200	17 hours	18 days
250	18 min	7,6 hours
300	39 sec	16 min
350	2,4 sec	59 sec
400	0,2 sec	5,4 sec

The values show that it is indeed correct to state that the full equilibrium concentration was not reached in the grain boundary sliding study. It is however likely that the P concentration in the grain boundaries was high enough to result in the impediment of sliding. If one applies eq. 14 in ref. 4. with the likely concentration of about 1500 ppm a back stress of about 15 MPa is obtained. This is enough to reduce the sliding by a factor of 10 in contradiction to the experimental observation that there is about an equal amount of sliding in OF and OFP copper.

4.3.2. What P concentration would be expected in an OFP copper grain boundary?

As noted above the expected initial P concentration in a grain boundary would be about 10-15 times the matrix concentration due to grain boundary movement in the temperature range 300-400 ˚C. The question then is: how much would it increase over time when the copper is stored at room temperature or slightly above. The answer is that it would almost not increase at all. A diffusion calculation shows that if the initial grain boundary concentration is 1500 ppm it will increase to 1630 ppm in 100 years if the copper is stored at 50 ˚C.

This result is fully consistent with the results of a recent micro analytical investigation at the department of Applied Physics at CTH [10]. Both OF and OFP copper were included in the investigation. The original intention was to use atom probe tomography (APT), a technique which makes it possible to map the distribution of foreign atoms in a microstructure. However, for reasons so far unknown the technique does not work well for copper [10]. Despite the problems it was possible to determine that the phosphorus was uniformly distributed in the matrix with a concentration in agreement with the known concentration, 80 wt-ppm, of the OFP material analysed. This shows that any segregation to the grain boundaries does not deplete the matrix of phosphorus, in agreement with the diffusion calculations above.

It was not possible to get a sample of APT which contained a grain boundary. Instead grain boundaries were analysed by TEM-EDS. Basically the grain boundary is oriented such that the electron beam is parallel with the grain boundary. With a beam width of 1 nm the prospects of determining even small grain boundary segregation would be quite good. However the electrons are scattered within the metal and the probed volume is much larger than that given by the beam width. The author made an estimate that the probed volume was about 10 nm [10]. Based on the detection limit for TEM-EDS of about 0.1 at% and that a monolayer (ML) of P in the grain boundary has a thickness of 0.1 nm the author concluded that $1/10^{th}$ of a monolayer would be detected which is well above the concentrations present according to the diffusion calculations.

To the present writer it seems more reasonable to assume that the grain boundary width is about 0.25 nm, the diameter of a copper atom, which would mean that a grain boundary concentration of $1/40^{th}$ or 2.5at% would be detectable. but 2.5at % is equal to 25000 ppm while expected grain boundary concentrations would be of the order of 1500 ppm. Thus the TEM-EDS result is consistent with the diffusion calculations.

4.3.3. Why is OFP copper not creep brittle when the grain boundaries slide as much as in the creep brittle OF copper?

A key observation in the grain boundary sliding study was that most of the tests on OF specimens run to failure had significantly lower failure strains than OFP specimens tested under the same conditions. Sandström and Wu comments on the results of the present writer as follows: “He attributed this behaviour to an unspecified embrittlement mechanism. At the high strain rates used, these results are not consistent with the model in Section 5” [4].

In the discussion of results of the grain boundary sliding study it is stated that according to the Sandström-Wu theory the OF specimens should have failed with

23% strain [4]. This is probably the basis for the statement that the results were inconsistent with the Sandström-Wu theory. However, subsequent calculations with the Sandström-Wu theory in the present study have shown that the statement on 23% strain was a mistake. In particular calculations have been made using the maximum stresses and failure times of the OF specimens and calculating which constant stress would be required in a creep test to result in the same failure time. The result is shown in Table 3

Table 3. Results of calculations of which constant creep stress (column 4) would give the same failure time as the tension tested OF specimens in the grain boundary sliding study [5].

Temperature ˚C	Time secs	Max stress MPa	Theory prediction MPa	Creep brittle or not?
200	25000	105	122	Yes
200	477	160	151	Yes
150	34000	145	150	Yes
150	682	200	164	Yes
100	455500	173	162	Yes
100	864	240	175	No
52	173000	245	174	No

The agreement between experiment and theory is strong enough to indicate that the OF specimens tested in the grain boundary sliding study failed by the same creep brittle mechanism as the OF specimens on which Sandström-Wu have based their theory.

Considering that the OF specimens failed by the creep brittle mechanism it becomes a problem that the OFP specimens tested under the same conditions were all ductile despite the fact that they had the same amount of grain boundary sliding as the OF specimens. In an attempt to solve the problem Sandström-Wu write “However, there was also a change in the work hardening with temperature. For this reason a function was fitted to the measured stress strain curves and the Considère criterion was used to predict the uniform elongation. It turned out that the measured uniform elongation could be predicted within 2% indicating that elongation values are controlled by the plastic deformation rather than by embrittlement.”

The Considere criterion is simply a criterion which tells when the maximum load occurs in a tension test based on current values of strain hardening and true stress. It is widely used to determine the onset of necking which is reasonably assumed, but not necessarily proven, to occur at the maximum load. This means that given a stress-strain curve the Considere criterion will automatically be fulfilled, and the Sandström-Wu analysis is meaningless. What is important is the cause of the reduction in strain hardening appearing in the stress-strain curves of the OF material. The view of the present writer is that that the loss in strain hardening is an apparent effect of cracking of grain boundaries, a view which is supported by fractographic evidence.

Sandström-Wu on the other hand seem to speculate that a pure face centered cubic (fcc) material like OF copper for some reason loses its normal strain hardening ability in a certain temperature-strain rate range without offering any reasonable explanation. Furthermore they must explain why an addition of a small amount of phosphorus removes this effect, but they have so far offered no explanation.

5. Conclusions

In a previous report [2] it was concluded that SKB has presented insufficient evidence to justify their position that the OFP copper has an adequate creep ductility during long term storage. This conclusion was based on the grain boundary sliding study which showed that the basis for the SKB theory on creep brittleness, necessary for extrapolation to storage conditions, was wrong. Recently SKB has attempted to invalidate the results of the grain boundary sliding study but in the present report it has been shown that the results on OF copper are consistent with their theory which has been calibrated with OF results from SKB. On the other hand SKB has failed to explain why OFP material having the same amount of grain boundary sliding as OF remains ductile under conditions when OF behaves in a creep brittle manner. Therefore the previous conclusion in [2] stands.

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2016:02

The Swedish Radiation Safety Authority has a comprehensive responsibility to ensure that society is safe from the effects of radiation. The Authority works to achieve radiation safety in a number of areas: nuclear power, medical care as well as commercial products and services. The Authority also works to achieve protection from natural radiation and to increase the level of radiation safety internationally.

The Swedish Radiation Safety Authority works proactively and preventively to protect people and the environment from the harmful effects of radiation, now and in the future. The Authority issues regulations and supervises compliance, while also supporting research, providing training and information, and issuing advice. Often, activities involving radiation require licences issued by the Authority. The Swedish Radiation Safety Authority maintains emergency preparedness around the clock with the aim of limiting the aftermath of radiation accidents and the unintentional spreading of radioactive substances. The Authority participates in international co-operation in order to promote radiation safety and finances projects aiming to raise the level of radiation safety in certain Eastern European countries.

The Authority reports to the Ministry of the Environment and has around 300 employees with competencies in the fields of engineering, natural and behavioural sciences, law, economics and communications. We have received quality, environmental and working environment certification.

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