## Metal, Ceramic and Composite Materials

Edited by Christian Coddet

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Selected, peer reviewed papers from the 2015 International Conference on Metal, Ceramic and Composite Materials (ICMCCM 2015),
January 24-25, 2015, Shanghai, China

Edited by

**Christian Coddet** 



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#### **Preface**

Dear Distinguished Authors and Guests,

The Organizing Committee warmly welcomes you to 2015 International conference on Metal ,Ceramic and Composite Materials (ICMCCM-2015) , 24<sup>th</sup>-25<sup>th</sup> , January, 2015, Shanghai, China.

The aim of ICMCCM 2015 is to present the latest research and results of scientists (professors, students, PhD Students, engineers, and post-doc scientist) related to Metal ,Ceramic and Composite Materials topics. This conference provides opportunities for the different areas delegates to exchange new ideas and application experiences face to face, to establish business or research relations and to find global partners for future collaboration.

After the peer-review process, the submitted papers were selected on the basis of originality, significance, and clarity for the purpose of the conference. The selected papers and additional late-breaking contributions to be presented as lectures will make an exciting technical program. The conference program is extremely rich, featuring high-impact presentation. We hope that the conference results constituted significant contribution to the knowledge in these up to date scientific field.

The proceeding records the fully refereed papers presented at the conference. The main conference themes and tracks are Metal, Ceramic and Composite Materials and other correlation technique. Hopefully, all participants and other interested readers benefit scientifically from the proceedings and also find it stimulating in the process.

On behalf of the organizing committee, I would like to especially thank Anne, Tanja, Dorthe and all the editors from Trans Tech Publications for their great support to ICMCCM 2015 .Without their excellent editorial work, ICMCCM 2015 will not be published so timely and successfully.

Finally we wish all the authors and attendees rewarding and enjoyable memory at ICMCCM 2015 in Shanghai, China. We look forward to your participation in the 2nd ICMCCM in 2016.

With our warmest regards,

Tony Sun

Conference Organizing Chair

February 3rd, 2015

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## **CHAPTER 1:**

Metal Materials, Alloys and Heat Treatment of Metals



## Effect of heat treatment on the creep properties of Zr-1Nb-0.12O nuclear cladding tubes

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Keywords: Fuel cladding tube, Creep properties, Zr-1Nb-0.12O, Heat treatment

#### Abstract

Creep properties of one-step and two-step annealed Zr-1Nb-0.12O cladding tubes were studied. Creep tests were carried out at 450~500 oC with the applied stress between 80MPa and 120MPa The creep rates of the two-step annealed Zr-1Nb-0.12O alloy were found to be slower than those of the one-step annealed Zr-1Nb-0.12O alloy. The creep rate decreased with increase of grain size with annealing for Zr-1Nb-0.12O at intermediate temperatures, suggesting the creep resistance can be enhanced by the grain size control. The creep life of two-step annealed Zr-1Nb-0.12O increased over the one-step annealed Zr-1Nb-0.12O by the factor of 18~20 despite the greater initial instantaneous strain.

#### Introduction

Zr alloy cladding tubes play important roles not only as the first barrier preventing nuclear fission products in the  $UO_2$  pellet from being released into the reactor coolant, but also as the conductor transferring nuclear fission-induced heat effectively to the coolant. From the viewpoint of the nuclear fuel integrity and the reactor safety [1-3], high temperature creep behavior of Zr cladding tubes along with corrosion behavior is one of the key performance parameters to be evaluated since they need to sustain the load arising from the pellet-clad mechanical interaction as well as the load caused by the reactor coolant over pressure acting on the outer surface of the Zr cladding tubes in the corrosive environment [2]

It was also found that the higher heat treatment temperature after the final pilgering step lowered the mechanical strength, but enhanced the corrosion and growth resistance [5]. Some cladding tubes are being heat treated in the stress relieved condition after cold pilgering to render the better mechanical strength and creep strength while other tubes are being heat treated in the recrystallized condition to render the better corrosion resistance and growth resistance at the expense of the mechanical strength. Experimental studies [4] suggest that the creep resistance can be modified not only by the addition or reduction of a certain alloying elements, but also by the grain size control in alloys. In this study, the effects of high temperature annealing and the resultant grain growth on the creep resistance of Zr-1Nb-0.12O cladding tubes were studied.

#### **Experimental Details**

In this study, Zr-1Nb-0.12O alloy cladding tubes were processed for studies of creep characteristics. Zr-1Nb-0.12O alloy cladding tubes were cold-pilgered with two intermediate heat treatments and finally annealed at 560oC for 5 hrs after final cold pilgering. Some cladding tubes were additionally annealed at 700oC for 3 hrs to induce the grain growth. The specimens additionally annealed at 700 oC will be called "two-step annealed" specimens. In order to examine the creep characteristics in the circumferential direction, the Zr alloy rings with the width of 4 mm were cut vertically against the axial direction of tubes. In this study, a special grip with two half-cylinders with 1 mm wide space between them was used to strain the ring specimen with maintaining a circular configuration of the ring in contact with the grips all the way during the tests [1-3,5]. Constant stress creep testers were used to investigate the effect of grain size on creep deformation behaviors. The creep tests were performed at 450, 475 and 500°C with the applied

stress between 80 MPa and 120 MPa. For microstructural analyses and fracture studies, transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were carried out.

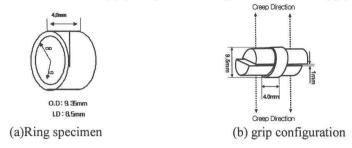


Fig. 1. Schematic configuration of (a) ring specimen and (b) grip for creep test

#### Results and Discussion

Grains of annealed Zr-1Nb-0.12O tubes at 560oC for 5 hrs were too small to be identified by a typical optical micrographic analysis. Fig.2 displays the typical TEM microstructure of the Zr-1Nb-0.12O alloy nuclear cladding tubes annealed at  $560^{\circ}$ C for 5 h (a) and additionally high temperature annealed at  $700^{\circ}$ C for 3 h (b). The regularly annealed (one-step) Zr-1Nb-0.12O tubes displayed small equi-axed grains with dislocations and dislocation tangles. This means that dislocation generation and storage also occurs during the pilgering process along with the continuous recrystallization. In the two-step annealed Zr-1Nb-0.12O tubes, dislocation-free recrystallized grains with a larger grain size than regularly annealed Zr-1Nb-0.12O tubes. The grain size of annealed and two-step annealed Zr-1Nb-0.12O tubes was observed to be  $\sim$ 1.2  $\mu$ m and  $\sim$ 5  $\mu$ m respectively.

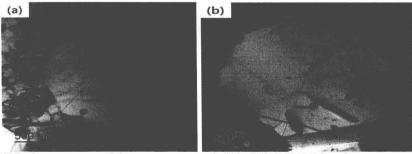


Fig. 2. TEM microstructure of the one-step annealed Zr-1Nb-0.12O (a) and two-step annealed Zr-1Nb-0.12O (b)

Fig. 3(a) and 3(b) exhibit creep curves of Zr-1Nb-0.12O cladding tubes; one-step annealed (a) and two-step annealed (b). Both one-step annealed and two-step annealed Zr-1Nb-0.12O alloy exhibit typical creep deformation curves although two-step annealed specimen exhibit less-pronounced primary period. It should be noted that the creep rates are slower in the two-step annealed alloy than in one-step annealed specimens at all loading condition. It should be emphasized that the primary creep period is shorter in the two-step annealed specimen despite the initially high instantaneous strain upon loading because of lower strength. It was shown that not only creep rate is faster but also creep life is shorter in the one-step annealed Zr-1Nb-0.12O than in the two-step annealed Zr-1Nb-0.12O. For example, the two-step annealed Zr-1Nb-0.12O fractured after 198 hrs at 120 MPa, 500oC whereas the one-step annealed counterpart fractured only after 11hrs under the same loading and temperature conditions. The creep life of two-step annealed Zr-1Nb-0.12O increased over the one-step annealed Zr-1Nb-0.12O by the factor of 18~20.

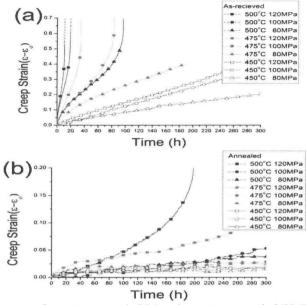


Fig. 3. Creep curves of one-step annealed (a) and two-step annealed (b) Zr-1Nb-0.12O.

Creep life has been suggested to increase with increase of grain size because the contribution of the fast diffusion along grain boundaries decreases. However, dislocation creep process has been considered to be grain size independent. For example, at intermediate temperatures where dislocation creep is dominant, that the creep rate of aluminium is grain size independent [6]. However, other studies have indicated that the creep rate may increase or decrease with decreasing grain size, or be grain size-insensitive in copper [6]. Lee et al. [7] reported that, in 316LN stainless steel, the creep rate decreased to a minimum value at the intermediate grain size of  $80 \sim 130~\mu m$  and then increased with further increase of grain size.

Lee et al.[7] suggested that their data agreed with Garofalo's model in which grain boundaries act simultaneously as both dislocation sources and barriers to dislocation movement. Generally, the grain boundaries which act as barriers reduce the steady state creep rate but they also sometimes accelerate it, being dislocation sources [6]. For copper and brass, the steady state creep rate is proportional to the square of grain size at low temperatures. The steady state creep rate of lead is proportional to the inverse of grain size at high temperatures. Wilshire and Palmer [6] suggested that the contribution of higher dislocation recovery and rearrangement within the boundary region would increase with decreasing grain size, resulting in the increase of creep rates with decrease of grain size. Both models suggest that the effect of grain size varies with experimental temperature and conditions even in identical materials and the dislocation activity near-grain-boundary regions could increase the creep rate in the small grain size region, supporting the decrease of creep rate with increase of grain size in the present study.

Fig. 5 show the SEM images of fracture surfaces of one-step annealed and two-step annealed Zr alloy crept at 100MPa and 475 °C. In both specimens, dimples were observed on the fracture surface. The presence of dimples suggests a typical ductile fracture in general. In the annealed specimen, some secondary cracks appeared to be formed along the grain boundaries. In general, with the increase of temperature, the number and mobility of thermal vacancies increase, causing more vacancies or impurities to diffuse to grain boundaries.

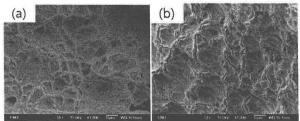


Fig. 5. SEM fracture surface images of Zr-1Nb-0.12O at 100MPa and 475 °C: (a) one-step annealed Zr alloy and (b) annealed Zr alloy.

#### Summary

Creep tests were performed on stress-relieved and annealed Zr-1Nb-0.12O and the results can be summarized as follows:

- 1. The creep rate decreased with increase of grain size with annealing for Zr-1Nb-0.12O at intermediate temperatures, suggesting the creep resistance can be enhanced by the grain size control.
- 2. The creep life of two-step annealed Zr-1Nb-0.12O increased over the one-step annealed Zr-1Nb-0.12O by the factor of  $18\sim20$ .
- 3. The primary creep period is shorter in the two-step annealed specimen despite the initially high instantaneous strain upon loading because of lower strength.

#### Acknowledgment

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