High Temperature Deformation Behavior of Nb-1Zr and Nb-1Zr-0.1C

R. Kapoor, A.N. Behera, A. Sarkar and J.K. Chakravartty
Materials Group

Abstract

Nb-1Zr-0.1C alloy is a potential candidate material for use in advanced nuclear reactors. An essential step in the fabrication of components is the thermo-mechanical processing of the alloy. These are performed at high temperatures, where either dynamic recovery or dynamic recrystallization occurs. The resultant microstructure is either refined or coarsened depending upon the applied strain rates and temperatures of hot working. Here the microstructural evolution of hot deformed Nb-1Zr and Nb-1Zr-0.1C alloys are presented and optimum domains of their hot working are identified. The microstructural features of dynamic recovery and recrystallization obtained during hot deformation are correlated to the corresponding stress-strain behaviour and strain rate sensitivity.

Introduction

Niobium alloys (melting point of ~ 2460 °C) offer attractive high-temperature properties suitable for applications in nuclear environments including high temperature strength, good thermal conductivity and compatibility with most liquid metal coolants [1-4]. Nb alloyed with 1%Zr adds to solid solution hardening and the addition of 0.1 wt% C results in very fine (Nb,Zr)C precipitates which enhances creep resistance [5]. Due to these properties, Nb-Zr alloys are being actively considered for use in high temperature applications in compact high temperature reactor (CHTR) [6]. Fabrication of these alloys into products with desired microstructure and free of volumetric defects requires optimum thermo-mechanical processing.

The usual route during industrial processing of materials is melting, casting, thermo-mechanical processing, and secondary processing steps like intermediate heat treatment, cold working, finishing operations, etc., resulting in the final product. Of these, thermo-mechanical processing is an important step where the cast microstructure breaks down and an intermediate defect free microstructure evolves. Thermo-mechanical processing (TMP) is carried out at high temperatures (usually around half the melting point and is also referred to as hot condition) with the material being deformed to large strains either in single or multiple steps. The two important independent parameters during TMP are the temperature (T) and strain rate (\(\dot{\varepsilon}\)) of deformation. Microstructural evolution during thermo-mechanical processing is dependent on the restoration mechanisms operating during those processing conditions, such as dynamic recovery, dynamic recrystallization and grain boundary deformation. Of these dynamic recrystallization (DRX) is an important restoration mechanism observed in almost all hot deformed metals and alloys. An overview of the hot deformation mechanisms can be found in References [7] and [8]. Presented here is the hot deformation behaviour and microstructural evolution of Nb-1Zr and Nb-1Zr-0.1C alloy deformed at temperatures from 700 to 1700 °C and strain rates of 3x10^{-3} to 1 s^{-1}. The basics of the method to determine the optimum hot working parameters are also discussed.

Determining the optimum hot workability

The optimum hot working conditions (T and \(\dot{\varepsilon}\)) are determined on a lab scale by carrying out compression tests over a range of temperatures and strain rates. Apart from flow stress (\(\sigma\)) being of prime concern during deformation (lower flow stresses imply easier deformation), the strain rate sensitivity is also important in determining the optimum hot working domain. The strain rate sensitivity \(m\) is the slope of the log \(\sigma\) vs. log \(\dot{\varepsilon}\) curve, i.e.

\[
m = \frac{\partial \log \sigma}{\partial \log \dot{\varepsilon}}
\]

A higher value of strain rate sensitivity ensures strain rate hardening, which delays necking in tension [9, 10] and prevents instabilities in compression [11]. The method of determining and mapping out \(m\) is shown in Fig. 1. The strain rate sensitivity is calculated from the \(\sigma-\dot{\varepsilon}\) data at large strain values (usually between 0.5 to 0.6) for various T and \(\dot{\varepsilon}\) as shown in Fig. 1(a) and (b). For each strain rate employed, the stress values
are interpolated at finely spaced log $\sigma$ and $1/T$ intervals. At each of the temperature values (including interpolated ones) the log $\sigma$ vs. log $\dot{\varepsilon}$ data is fitted to a cubic polynomial as seen in Fig. 3(c) and the stress values interpolated at finely spaced log $\dot{\varepsilon}$ values. These two interpolations produce a grid of stress values over the entire strain rate and temperature range of tests. The strain rate sensitivity is calculated by taking the derivative at each point of the log $\sigma$ - log $\dot{\varepsilon}$ curve (eq. 1). These calculated values of $m$ are plotted for each grid point on the $T$ and log $\dot{\varepsilon}$ space resulting in a 3D plot seen in Fig. 1(d). This is then translated into a 2-D contour map as shown in Fig. 1(e). Such maps show domains of high $m$ (as shown in red) and regions of low $m$ (as shown in blue). The contours join points of equal $m$ values, making them iso-strain rate sensitivity contours. The microstructure at different conditions of temperature and strain rate are then observed and correlated with the flow stress and strain rate sensitivity. There are numerous such studies where the strain rate sensitivity is computed and plotted as a contour map (e.g., [12-15]). Apart from identifying regimes of high $m$, regimes of instability and flow localization also need to be identified and avoided during processing. In literature there are several approaches for identification of regimes of instability during deformation, based on work-hardening/softening [11] and extremum principles [16]. However, a conservative condition for the occurrence of instability in these methods is that the value of $m$ reduces to $m < 0$. There are other less stringent conditions for instability, but some of these are controversial and will not be discussed here. As an example Ref. [15] discusses these instability conditions for the specific case of cast Zr-2.5Nb.

**Hot deformation of Nb-1Zr and Nb-1Zr0.1C**

The hot deformation behaviour and microstructural evolution of Nb-1Zr and Nb-1Zr0.1C were studied using compression tests carried out in vacuum to a true strain of 0.6 in the temperature range of 700 to 1700 °C and the strain rate range of $10^{-3}$ to 10 s$^{-1}$. The flow stress of Nb-1Zr0.1C was higher than that of Nb-1Zr as seen in Fig. 2 for two strain rates of 0.1
For example, at 1000 °C and 1 s\(^{-1}\), Nb-1Zr-0.1C had a flow stress of 250 MPa with moderate strain-hardening, whereas Nb-1Zr for the same condition showed a low yield stress and a substantial work-hardening but to lower stress values. At higher temperatures between 1500 to 1700 °C, both alloys showed steady state behaviour but the flow stress of Nb-1Zr-0.1C was still higher than that of Nb-1Zr.

The high strain rate sensitivity domain of Nb-1Zr occurred at lower temperature (1500 °C) as compared to that of Nb-1Zr-0.1C (1700 °C) as seen in Fig. 3. Nb-1Zr-0.1C showed a high \(m\) domain from 1500 to 1700 °C and \(3\times10^{-3}\) to \(0.1\) s\(^{-1}\), whereas Nb-1Zr showed it in the range from 1300 to 1500 °C and \(10^{-2}\) to \(1\) s\(^{-1}\). Addition of carbon to Nb-1Zr is known to result in formation of \((\text{Nb},\text{Zr})\)C which is a stable phase. This carbide formation shifts the high \(m\) domain to higher temperatures. For both Nb-1Zr and Nb-1Zr-0.1C, the iso-strain rate sensitivity contour maps show different domains and strain rates.
alloys, the low strain rate sensitivity domain appeared at lower temperatures and higher strain rates.

**Microstructural comparison**

Fig. 4 shows a comparison of electron back scatter diffraction (EBSD) maps of Nb-1Zr and Nb-1Zr-0.1C alloys deformed respectively at temperature of 1500 °C for strain rates of 0.01 and 0.1 s⁻¹ and at 1600 °C for strain rate of 0.1 s⁻¹. For both alloys, at strain rate of 0.01 s⁻¹ and temperature of 1500 °C grains appeared recrystallized and nearly equiaxed. At this condition, grains of Nb-0.1Zr-0.1C are finer as compared to that of Nb-1Zr alloy. For strain rate of 0.1 s⁻¹ at 1500 °C Nb-1Zr-0.1C showed elongated grains whereas Nb-1Zr showed equiaxed grains. At this condition dynamic recrystallization appeared to have started but remained incomplete for Nb-1Zr-0.1C alloys whereas it appeared completed for Nb-1Zr. At strain rate of 0.1 s⁻¹ DRX microstructure results from the initial grains which get elongated during deformation for Nb-1Zr-0.1C alloys at 1500 and 1600 °C. Decreasing temperature and increasing strain rate resulted in grain refinement.

**Effect of carbon addition**

The role of zirconium and carbon in Nb is to strengthen the material through solid solution and precipitation, respectively. The stress - strain rate data at 900 and 1000 °C of Nb [17], Nb-1Zr and Nb-1Zr-0.1C [14] is shown in Fig. 5. It is seen that Nb and Nb-1Zr lie in the same stress range and lower to that of Nb-1Zr-0.1C. This shows that the solid solution effect of Zr is insignificant as compared to that hardening produced by carbide precipitates. The activation energies for the deformation of Nb and Nb-1Zr alloys are 245 and 260 kJ/mol respectively, while that of Nb-1Zr-0.1C is 360 kJ/mol. A higher activation energy suggests that for same conditions the deformation is more difficult. Further, as the activation energy for self-diffusion of Nb is 400 kJ/mol, it implies that for Nb and Nb-1Zr (\(Q < Q_{SD}\)) dislocation based core diffusion may be the rate controlling mechanism.

**Conclusions**

A comparison of the hot deformation behaviour of Nb-1Zr and Nb-1Zr-0.1C showed the following.

1. Nb-1Zr-0.1C shows a higher flow stress as compared to Nb-1Zr.
2. Both Nb-1Zr and Nb-1Zr-0.1C dynamically recrystallizes during hot deformation.

---

**Fig. 4:** EBSD maps of Nb-1Zr and Nb-1Zr-0.1C deformed at 1500 °C 0.01 and 0.1 s⁻¹ and 1600 °C and 0.1 s⁻¹ up to a strain of 0.6. The colours represent the crystal orientation according to stereographic triangle with respect to normal of polishing surface.

**Fig. 5:** Flow stress vs. strain rate for Nb [17], Nb-1Zr and Nb-1Zr-0.1C [14].
3. The temperature of dynamic recrystallization of Nb-1Zr is lower as compared to Nb-1Zr-0.1C (1300 to 1500 °C vs. 1500 to 1700 °C).
4. Nb-1Zr showed equiaxed grains indicative of a completed dynamic recrystallization process whereas Nb-1Zr-0.1C showed elongated grains with fine grains at grain boundaries (necklace structure) indicative of start but incomplete dynamic recrystallization process.

References