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Multi-temperature indentation creep tests on nanotwinned copper

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ABSTRACT:

The present work further develops the multi-temperature approach on load, time, and temperature-dependent deformation for indentation creep. Multi-temperature micro-indentation creep tests were carried out on nanotwinned copper (nt-Cu) at five temperatures of 22 °C (RT), 40 °C, 50 °C, 60 °C and 70 °C. In analogy with stress, hardness is used to gauge the indentation creep loading level, while the indentation depth is used to characterize the indentation creep deformation and the creep strain rate is represented by the indentation depth strain rate. The multi-temperature micro-indentation creep tests generate sufficiently large experimental data, which makes the development of a novel formula for indentation creep feasible. There are few intrinsic parameters that characterize the capability of the microstructure of a material against load, time, and temperature dependent deformation and they are the strain rate sensitivity, the athermal hardness exponent, intrinsic activation energy, and activation volume. The strain rate sensitivity is determined from isothermal creep data at one temperature, while the other parameters have to be determined from multi-temperature creep data. The novel formula is validated by the experimental data of the multi-temperature indentation creep tests on the nt-Cu. The creep mechanisms of the nt-Cu are also discussed and analyzed by using the determined values of the intrinsic parameters.

1. Introduction

Creep behaviors of materials under sustained load at elevated temperatures are essential to their practical applications. The rate of creep deformation is related to the types of materials, and the external conditions including load, creep time, and temperature. Based on experiments, atomistic simulations and/or theoretical models, the creep deformation behaviors and associated mechanisms of various types of materials, such as metals and alloys (Kassner and Pérez-Prado, 2004; Lee et al., 2016), polymer and composites (Jia et al., 2011; Tehrani et al., 2011), and ceramics (Gan and Tomar, 2010; Kassner et al., 2007), etc., have been extensively investigated for many years. In the conventional polycrystalline metals, the creep deformation can be accommodated by the lattice or grain boundary (GB) diffusion (Choi et al., 2013a; Coble, 1963; Fischer and Svoboda, 2011; Herring, 1950), and movements and associated consequences of interfaces, GBs and twin boundaries (TBs) (Basirat et al., 2012; Morra et al., 2009; Muñoz-Morris et al., 2009;

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Oberson and Ankem, 2009), and dislocation activities (Wang et al., 2011), etc.

When grain size is at the nanoscale (typically < 100 nm) in polycrystalline metals, the nanograined (ng) metals exhibit enhanced strength. However, the creep deformation is severely accelerated in the ng metals, because decreasing grain size significantly increases the volume fraction of GBs, which possess very poor thermal stability and thus provide fast diffusion path for creep deformation and failure (Sanders et al., 1997; Wang et al., 2012b, 2013b). The creep rate in ng metals depends greatly on the grain size, that is, the smaller the grain size is, the higher the creep rate will be (Sanders et al., 1997). Similarly, the reported uniaxial creep strains and creep strain rates in smaller Ni nano-pillars were correspondingly higher than those in Ni micro-pillars (Choi et al., 2013a) due to surface diffusion. Therefore, the weak creep resistance of ng metals is one of the bottleneck problems for their practical applications, especially at elevated temperatures. In contrast, the resistance of coherent twin boundaries (CTBs) against dislocation motion is much more moderated, thereby optimizing both the strength and ductility of metals (He et al., 2016; Lu et al., 2004; Xiao et al., 2015). CTBs are not fast diffusion channels in comparison with GBs, resulting in more thermal stability of nanotwinned (nt) structures compared with the twin-free ng counterparts (Li et al., 2016; Zhang and Misra, 2012; Zheng et al., 2013). For example, the cyclic nano- and micro-indentation creep (Bezares et al., 2012) and uniaxial tensile creep experiments (Yang et al., 2016b) on nt-Cu indicate that nt structure exhibits greater creep resistance than twin-free ng-Cu, and the simulations (Jiao and Kulkarni, 2015) reveal that the creep resistance increases with decreasing TB spacing.

Nano/micro-indentation is a fast and effective technique to characterize the mechanical properties of materials at the nanometer/micrometer scales. Due to its unique feature of small size-scaled characterization, the nano/micro-indentation has been widely utilized in the mechanical characterization of various materials, especially nanomaterials, nanostructured materials, biomaterials, thin films (Ma et al., 2012; Wang et al., 2015) etc., Within such small detected volume in nano/micro-indentation tests, the strain magnitude, the stress level, and the strain rate could be considerably high, which are hardly achieved in conventional tensile tests (Choi et al., 2013b). In normal uniaxial tensile (compression) test, the cross-head movement rate of a testing machine is pre-set and the load cell and the extension meter record, as functions of time, the load and sample displacement, respectively. Then, based on the geometry of a tested sample, load and displacement are correspondingly converted to stress and strain, and then stress rate and strain rate can be calculated. The strain rate is linked to the cross-head movement rate and a pre-set cross-head movement rate corresponds generally to a constant strain rate. In this sense, normal uniaxial tensile (compression) test is carried out under a sustained strain rate, during which the stress varies with the deformation characterized by strain. In contrast, a sustained stress is applied on a sample in uniaxial tensile (compression) creep test and creep deformation strain is recorded with creep time. Based on the creep deformation behavior, creep deformation is usually divided into three stages of the primary (transient) stage, the secondary steady stage, and the tertiary (unstable) stage. In the primary stage, the strain rate decreases with time and reaches a steady value in the secondary stage, while in the tertiary stage, the strain rate increases until failure of the crept sample. The creep strain rate in the secondary steady stage is approximately constant independent of time, which is the most critical creep strain rate. This is because that the period of steady state creep spends, in most cases, the largest portion of the creep life, and the higher the steady creep rate is, the shorter the survival life of the crept material will be. In indentation creep tests there is a challenging issue that which parameters should be used to represent and describe the loading level and the degree of deformation. This is because the stress distribution is completely inhomogeneous inside the tested sample under an indentation load (Wang et al., 2015) and the inhomogeneous stress distribution causes inhomogeneous deformation. It is suggested that hardness might be the appropriate parameter to describe the loading level, since hardness in nanoindentation tests is defined as the averaged pressure on the projected contact area. Indentation depth might be the appropriate parameter to describe the deformation, because indentation depth is the only measurable parameter in nanoindentation creep under a given indentation load (Gan and Tomar, 2011). Indentation creep is actually the inhomogeneous deformation varying with time under a given load, in which the indentation depth increases and the hardness decreases monotonically with time. Commonly, the creep deformation will eventually saturate under a given compressive indentation load (Zhang et al., 2017a).

Sharma et al. (2005) conducted the indentation creep tests on the iron aluminide intermetallic (Fe-28Al-3Cr) at the temperature range of 843–963 K. The experimental results showed that the hardness decreased with creep time and the stress exponent n was weakly dependent on temperature, being 5.52 at 843 K and 4.53 at 963 K. The determined stress exponent of ~ 5 and the activation energy in the range of 339–341 kJ/mol (Sharma et al., 2005) were found to be consistent with the dislocation climb creep mechanism. Nanoindentation creep tests on ng Cu-based alloys at room temperature (RT) (Liu et al., 2012) exhibited that the stress exponents (ranging from 5 to 50) increased with the increase of the indentation load. Besides, it was found that the higher the temperature was, the lower the determined activation volume and stress exponent would be (Alizadeh et al., 2013; Ma et al., 2002; Ranganath and Mishra, 1996; Wang et al., 2009a). Wang et al. (2009a) performed the indentation creep tests on the ng-Ni with grain size of 14 nm under sustained load of 1 mN at temperatures of 348 K, 398 K and 448 K, respectively. The corresponded stress exponents and activation volumes at the three temperatures were determined to be 14.81 and $3.78b^3$, 7.14 and $2.74b^3$, and 4.91 and $2.48b^3$, respectively, where b denoted the magnitude of Burgers vector. The calculated activation energy of 123.1 kJ/mol was remarkably close to that for the GB self-diffusion in Ni, but far less than that for lattice diffusion, indicating that the rate controlling process was meditated by GB diffusion in the ng-Ni (Wang et al., 2009a). Zhang et al. carried out the indentation creep tests on In-617 alloy under sustained load of 200–400 mN at temperatures from RT up to 800 °C (Zhang et al., 2017a) and Raman spectroscopy measurements of depth sensitive stress distribution (Zhang et al., 2017b). The determined stress exponent values for the indentation creep were between 5 and 6, thereby suggesting that the creep mechanism of IN-617 at nano/micro scales was dominated by dislocation climb. The creep properties of silicon microcantilevers were in-situ investigated at temperatures of RT, 50 °C and 100 °C under uniaxial compressive stress within range of 50–100 MPa (Gan et al., 2014), showing that the creep rate of the silicon cantilever was increased with both compressive stress and temperature.

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