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Room temperature deformation in the Fe_7Mo_6 μ-Phase

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ABSTRACT

The role of topologically close packed (TCP) phases in deformation of superalloys and steels is still not fully resolved. In particular, the intrinsic deformation mechanisms of these phases are largely unknown including the active slip systems in most of these complex crystal structures. Here, we present a first detailed investigation of the mechanical properties of the Fe_7Mo_6 μ-phase at room temperature using microcompression and nanoindentation with statistical EBSD-assisted slip trace analysis and TEM imaging. Slip occurs predominantly on the basal and prismatic planes, resulting also in decohesion on prismatic planes with high defect density. The correlation of the deformation structures and measured hardness reveals pronounced hardening where interaction of slip planes occurs and prevalent deformation at pre-existing defects.

1. Introduction

Topologically close packed (TCP) phases, such as the μ-phase, form as precipitates in a wide range of materials. They are highly ordered phases both in terms of chemistry and stacking, although a wide range of compositions may be found (Cheng et al., 2012; Huhn et al., 2014). Their complex crystal structure usually results in pronounced brittleness of these phases (Sauthoff, 1995). The most prominent cases, where TCP phases form as precipitates, occur in heavily alloyed nickel- and iron-based superalloys, where TCP phases form either in the γ-γ′-matrix or the secondary reaction zone underneath a thermal barrier coating (Chen et al., 1980; Darolia et al., 1988; Rae and Reed, 2001; Walston et al., 1996; Yulin and Yunrong, 1982). TCP phases are further observed during joining, particularly in the presence of refractory metals such as in steel-tungsten joints implemented in fusion reactors (Matějíček et al., 2015).

Although it is commonly assumed that the μ-phase, like the other TCP phases, is detrimental to the mechanical properties and oxidation behaviour of superalloys, the underlying mechanisms are not yet fully understood and controversially discussed (Chen et al., 1980; Cheng et al., 2011; Epishin et al., 2010; He et al., 2005; Moverare et al., 2009; Pessah et al., 1992; Qin et al., 2009; Simonetti and Caron, 1998; Sims et al., 1987; Sugui et al., 2010; Tawancy, 1996; Wang et al., 2010, 2014; Yang et al., 2006; Zhao and Dong, 2012). In particular, it is not clear whether a certain amount of μ-phase precipitates might even be tolerated by the material without significant negative effects (Pessah et al., 1992; Simonetti and Caron, 1998).

In superalloys, the TCP μ-phase causes depletion of the γ-matrix in the solution strengthening refractory metal elements (Pessah et al., 1992; Simonetti and Caron, 1998; Sims et al., 1987; Yang et al., 2006). Dislocation pile-ups at intermetallic precipitates have been reported to cause microcracking or decohesion, embrittlement and eventually crack initiation (Chen et al., 1980; Cheng et al.,

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2011; Pessah et al., 1992; Qin et al., 2009; Simonetti and Caron, 1998; Tawancy, 1996; Zhao and Dong, 2012). Further, TCP phase precipitates at grain boundaries are thought to cause high stress concentrations (Pyczak et al., 2000; Sugui et al., 2010; Zhao and Dong, 2012) resulting from the high hardness of the μ-phase compared with the surrounding matrix but also the precipitates' shape and orientation relationship to the matrix. Spherical precipitates have been reported to be prone to decohesion, while needle- or plate-like precipitates have been found to lead to dislocation pile-ups and cracking (Pessah et al., 1992; Simonetti and Caron, 1998; Zhao and Dong, 2012). On the other hand, this can be inhibited due to good interfacial strength towards the matrix or a γ′-film enclosing μ-phase particles (Simonetti and Caron, 1998; Wang et al., 2010; Yang et al., 2006). During creep of γ-γ′-superalloys, μ and other TCP phase precipitates have been observed to accelerate rafting (Zhao and Dong, 2012) and to distort the unrafted (Pyczak et al., 2000; Yang et al., 2007) and, more severely, rafted microstructures (Han et al., 2008; le Graverend et al. (2011); Moverare et al., 2009; Simonetti and Caron, 1998; Sugui et al., 2010).

However, while in most studies the μ-phase precipitates are assumed not to deform themselves, ductility of needle shaped precipitates has indeed been found (Qin et al., 2009; Yang et al., 2006) and no detrimental effect on the tensile strength was found during high temperature tensile testing (Chen et al., 1980; Tawancy, 1996).

Particularly, the mechanisms by which μ-phase precipitates affect creep at high temperatures are still under discussion (Cheng et al., 2011; Epishin et al., 2010; Han et al., 2008; He et al., 2005; Moverare et al., 2009; Simonetti and Caron, 1998; Sugui et al., 2010; Volek and Singer, 2004; Wang et al., 2010; Yang et al., 2006). Le Graverend et al. (le Graverend et al., 2011) have conducted FEM calculations of nickel-based superalloys considering plasticity of μ-phase precipitates at higher temperatures. However, they (le Graverend et al., 2011) were not able to exactly replicate the crystal rotations of the γ′-rafts and μ-phase needles evolving from the local stress field of matrix and precipitates. Their simulations show indeed an influence in the microstructure evolution when considering plasticity and anisotropy of μ-phase precipitates. Given the lack of mechanical data on individual slip systems, the authors therefore have assumed deformation driven by stacking fault and twin formation observed elsewhere (Carvalho et al., 2000; Qin et al., 2009; Stenberg and Andersson, 1979; Tawancy, 1996). However, no quantitative information regarding critical resolved shear stresses (CRSS) on a given system are available from these studies. The anisotropy assumed in the constitutive model may therefore be incomplete or incorrect and lead to the deviation of the simulated microstructure evolution from experimental results reported (le Graverend et al., 2011).

Therefore, knowledge of the active slip systems, the underlying dislocation mechanisms and their critical resolved shear stresses is required to understand plasticity in TCP phases and other complex crystals as well as their effect on the co-deformation in alloys with intermetallic reinforcement. The deformation behaviour of the μ-phase is still largely uninvestigated (le Graverend et al., 2011), mainly due to its pronounced brittleness at ambient temperatures. The complexity of the μ-phase's crystal structure impedes a direct identification by estimates from the unit cell, for which not even deformation of the individual building blocks has been fully understood (Chisholm et al., 2005; Krämer and Schulze, 1968; Livingston and Hall, 1990).

The hexagonal unit cell of the μ-phase is given in Fig. 1a (Momma and Izumi, 2011) using the structural model by Lejaeghere et al. (2011). It exhibits a complex crystallographic structure built up from Frank-Kasper polyhedra (Frank and Kasper, 1958, 1959) of CN12, CN15, CN16 and CN15 packing (Kumar et al., 1998; Wagner et al., 1995), resulting in an alternating stacking of Zr_4Al_3 and Laves-phase triple layers. The space group of the μ-phase is 166 $R\bar{3}m$, with 13 (or 39 in hexagonal notation) atoms per unit cell (Cieslak et al., 2014; Krishna et al., 2013; Rae et al., 2000). Its typical stoichiometry is A_7B_6 and the archetype is Fe_7W_6 (Forsyth and D'Alte da Veiga, 1962; Magneli and Westgren, 1938). Smaller A atoms are positioned in CN12 polyhedra whilst larger B atoms occupy the higher coordinated ones (Cieslak et al., 2014; Kumar et al., 1998). Within a wide range of binary and ternary alloys of early and late transition metals (Carvalho et al., 2000; Forsyth and D'Alte da Veiga, 1962; Huhn et al., 2014; Matějíček et al., 2015; Ren et al., 2014; Wagner et al., 1995), the structural parameter **a** extends between 4.723 and 4.769 Å and **c** between 25.48 and 25.83 Å (Magneli and Westgren, 1938; Rae et al., 2000; Stenberg and Andersson, 1979).

Only a few recently published papers focus on the mechanical properties of the μ-phase itself, either examined by nanoindentation of precipitates within a creep deformed nickel-base superalloy (Rehman et al., 2015) and Fe-W composites (Matějíček et al., 2015), or by ab-initio assessment (Huhn et al., 2014; Ren et al., 2014) of different alloy systems. However, attempts have been made neither to identify the experimentally observed slip systems, nor to determine the CRSS of these. The defects described so far (Carvalho and De Hosson, 2001; Carvalho et al., 2000; Hiraga et al., 1983; Hirata et al., 2006; Qin et al., 2009; Stenberg and Andersson, 1979; Tawancy, 1996) are all growth related and not induced by deformation.

In this work, the dominant experimental difficulty of brittle failure before significant plastic deformation at low temperatures is overcome by employing nanoindentation in conjunction with atomic force microscopy, electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). This combination allows the identification of active slip planes in this complex crystal and can therefore not only give a better indication of the mechanical anisotropy but also guides subsequent uniaxial tests to determine critical resolved shear stresses (CRSS) on particular slip planes (Korte-Kerzel, 2017; Korte and Clegg, 2012). Such tests were carried out using single crystalline micropillars, in which brittle failure is largely suppressed even in the most brittle and complex crystals (Korte-Kerzel, 2017; Korte-Kerzel et al., 2018), and selected orientations giving high resolved shear stresses on the slip planes identified by indentation and TEM.

2. Experimental methods

2.1. Sample preparation

The sample composed of 45 wt.-% Fe and 55 wt.-% Mo was arc-melted from pure elements and subsequently re-melted and