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Deformation mechanisms during high temperature tensile creep of Ti_3AlC_2 MAX phase

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ABSTRACT

The deformation mechanisms involved in the tensile creep of a Ti_3AlC_2 specimen deformed, at 900 °C, to a 7.5% final strain are investigated through SEM and TEM observations. Tensile creep strain rate analyses on Ti_3AlC_2 deformed at 900 °C enabled to identify a Norton's law with a n coefficient around 2, suggesting that creep mechanisms are controlled by grain boundary sliding. TEM observations revealed a highly heterogeneous microstructure consisting in both grains without any dislocations and grains highly defected. This intragranular deformation involves three different microstructural features: dislocations mainly confined in the basal planes and possibly organized in hexagonal networks, numerous stacking faults, and original lenticular non planar defects that likely play an important role in the sample deformation. The deformed microstructure observed and the estimated Norton coefficient suggest that intergranular deformation play an additional important role in the deformation mechanisms.

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1. Introduction

In the past two decades, studies upon MAX phases, initially discovered by Nowotny [1] in the late 60's, have been spreading. These ternary nano-layered compounds can be described by the empirical formula $M_{n+1}AX_n$, with n = 1, 2 or 3, M an early transition metal, A an A-group element (i.e.: from columns IIIA to VIA of the periodic table) and X carbon and/or nitrogen. The three elements crystallize in a hexagonal P63/mmc lattice characterized by M_6X octahedra separated by A atomic layers. Their unique structure, combining both strong covalent M-X bonds and weaker M-A bonds, confers to MAX phases properties of both classes of materials. Like ceramics, they are stiff, lightweight, chemically stable and oxidation resistant; like metals, they are relatively soft, machinable, resistant to thermal shock and they exhibit good electric and thermal conductivity and good damage tolerance [2–4]. Wang et al. proposed a more specific review on Ti_2AlC and Ti_3AlC_2 [5].

MAX phases exhibit a Brittle-to-Ductile Transition (BDT) at high temperatures. This phenomenon has been mainly observed in Ti_3SiC_2 through compressive [6] and tensile tests [7]. In both cases, authors pointed out the strong dependence between the applied strain rate and the mechanical response of the MAX phase. Only few studies were performed on Ti_3AlC_2. Recently, compression tests performed by Zhang et al. [8] confirmed the existence of such a strain rate/mechanical behavior dependence in Ti_3AlC_2.

Deformation mechanisms were originally identified in Ti_3SiC_2 deformed at room temperature (RT) [9,10]. These studies suggest, as a result to high crystal lattice anisotropy, that dislocations are confined in the basal plane and therefore, deformation occurs through dislocations glide in [0001], delamination and kink band formation. Basal planes dislocations can be either single dislocations or dislocations arranged in arrays or walls. These microstructural features were also encountered in Ti_2AlN [11], Ti_4AlN_3 and Ti_3AlC_2 [12] after compression tests. More recently, Transmission Electron Microscopy (TEM) characterizations gave microstructural evidence of dislocations interactions like dislocation dipoles, nodes and networks lying in the basal plane in Ti_2AlN deformed under confinement pressure [11].

Only few TEM observations have been performed on MAX phases after high temperature testing (i. e. over the brittle-to-ductile transition temperature). In several studies [8,13], the BDT is assumed to involve activation of out-of-basal slip systems.

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Indeed, Guitton et al. showed that compression tests performed on Ti_2AlN at 900 °C lead to the activation of pyramidal or prismatic slip systems [13]. Recently, Zhang et al. confirmed these results through observation of Ti_3AlC_2 samples deformed under uniaxial compression tests performed in the temperature range 1000–1200 °C [8]. The authors assign the observed strain hardening to hexagonal dislocation networks compensated by initiation of non-basal plane dislocation movement leading to a remarkable plasticity. However, the understanding of this phenomenon is still confused since only basal plane dislocations were observed by Barcelo et al. in Ti_3SiC_2 after tensile creep tests at 1000 °C [14]. These observations support Barsoum's interpretation of the BDT as a phenomenon in which internal stresses are likely to be accommodated by interlaminar decohesion, grain bending or kinking, and/or grain boundary decohesion and sliding [4].

To our best knowledge, very few papers deal with tensile creep properties of MAX phases. Tallman et al. studied Ti_2AlC tensile creep in the 1000–1150 °C temperature range and 10–40 MPa stress range [15]. Secondary creep regime can be described by a Norton-Bailey law $\dot{\varepsilon}_{min}(s^{-1}) = \varepsilon_0 A\left(\frac{\sigma}{\sigma_0}\right)^n \exp\left(\frac{Q}{RT}\right)$ where n = 2.5 ± 0.3 and Q = 362 ± 88 kJ/mol. Fine and coarse-grained Ti_3SiC_2 samples [16,17] also obey a Norton-Bailey law in the 1000–1200 °C temperature range and 10–100 MPa stress range with n = 1.5 ± 0.1 and Q = 445 ± 10 kJ/mol for fine-grained microstructure and n = 2.0 ± 0.1 and Q = 458 ± 12 kJ/mol for coarse-grained microstructure. Creep experiments generate large internal stresses that dissipate through a combination of delamination, grain boundary decohesion and triple points cavitation. In the three tensile creep studies mentioned above [15–17], main deformation mechanism is assigned to dislocation creep. The little impact of grain size on minimum creep rate of Ti_3SiC_2 indicates that grain boundary sliding cannot be the dominant mechanism involved in creep even though it participates to the accommodation of the high internal stresses in the first two stages of creep. TEM observations on post creep Ti_3SiC_2 microstructure indicate well defined (0001) dislocations glide and dislocation walls of same sign or opposite sign as the main plastic deformation mechanisms identified in individual grains during creep [14].

The present paper reports the first high temperature tensile creep experiments performed on Ti_3AlC_2. It focuses on the deformation mechanisms involved during creep of the specimen at 900 °C through TEM investigation.

2. Experimental details

Highly dense Ti_3AlC_2 samples were synthesized by powder metallurgy. Titanium, aluminum and titanium carbide powders were blended in quasi stoichiometric proportions (i.e. 1.9 TiC-1.05 Al-Ti) for 20 min in a Turbula® mixer. The reactant powder mixture is encapsulated in a glass container under vacuum and then, sintered under argon atmosphere during 2 h at 1450 °C to produce highly porous Ti_3AlC_2 bulk material. Such a porous material is then crushed into Ti_3AlC_2 powder. Finally, Ti_3AlC_2 powder is densified by Spark Plasma Sintering (SPS) under vacuum.

Electric discharge machining process is used to cut specimens from the bulk materials. In this work, 2 geometries are tested: specimens with cylindrical gauge length are used for lower stresses tensile creep tests and 2 flat parts are machined on gauge length to be tested at higher stresses to lower the stress concentration in specimens' grip area (threaded heads). Specimens are then polished along the stress axis with SiC paper up to 2400 grade prior to creep tests. In this study, tensile creep tests are performed at 900 °C and 1000 °C (±1 °C temperature variation along the gauge length) in air for stresses in the range 45–210 MPa. The specimens have been tested using a test bench equipped with a resistive furnace and grips made of Mar-M200 directionally solidified superalloy [18]. Before applying the mechanical load, the temperature is stabilized for 3 h to avoid any further thermal strain measurement. Creep strain is then measured with a Linear Variable Displacement Transducer (LVDT) that follows the displacement of the specimen grip areas.

Identification of impurities contained in the bulk material is performed through X-Ray Diffraction analyses (XRD, Bruker D8 using Cu-Kα rays) coupled to Rietveld refinement using MAUD software [19]. Microstructures of bulk material and post-creep specimens are investigated by Optical Microscopy (OM, Zeiss Imager Vario Z2), Scanning Electron Microscopy (SEM) observations coupled to Energy Dispersive X-ray Spectroscopy (EDXS) analyses (Jeol JSM 7001F TTLS equipped with back-scattered electrons detector and Oxford Instruments X-MAX detector for EDXS analyses). After creep deformation and/or failure, specimens are cut parallel to the applied load, embedded in a conductive mounting resin and polished up to 1 μm polishing cloth prior to observations. A creep test performed at 900°C/140 MPa was interrupted at 7.5% strain in order to prepare TEM thin foils. Two samples are sliced in the specimen: one is sliced perpendicular to the applied load and another one at 45° from the applied load. The two samples are thinned by a slow mechanical polishing down to few microns and observed areas within thin foils were prepared using a Precision Ion Polishing System from GATAN. TEM observations are performed on a Philips CM 20 microscope under 200 kV.

3. Results

After SPS process, Rietveld refinement of the XRD pattern gives evidence for the presence of less than 2 wt.% of titanium carbide in the sintered sample. SEM observations coupled with EDXS analyses enabled to confirm the presence of TiC and give also evidence for the presence of small amount of Ti_xAl_y intermetallics (variable x and y contents) and alumina (Al_2O_3) grains randomly distributed at Ti_3AlC_2 grain boundaries (Fig. 1a and Fig. 1b). The presence of a large amount of impurities in the areas observed in Fig. 1a and 1b (between 1.7% and 2.3% of the surface fraction for alumina, about 2.4% for TiC and below 2.7% for Ti_xAl_y intermetallics) is due to this random distribution of the inclusions but is not representative of the bulk material, as impurities content is remaining low. A quantitative study has been conducted on an extended area representative of the final microstructure (Fig. 1c) using ImageJ software in order to assess inclusions contents in the sample. The alumina content assessed by this method is below 2.5% (Al_2O_3 and pores contents are here jointly quantified due to their similar contrasts) which is consistent with the results extracted from Fig. 1a and 1b. White light OM micrograph enables to assess both TiC and Ti_xAl_y contents which are respectively close to 1.06% and 0.15% of the surface fraction. Thus, the high temperature softening role played by intermetallics located at triple points and grain boundaries will not be considered in this study due to their small content.

The final fine-grained microstructure is also qualitatively characterized by a large Ti_3AlC_2 grain size distribution which contains both grains of a few μm in width and dozens of μm in length and sub-micron grains as it can be seen in Fig. 1c. Besides, most of the grains exhibit a high elongated shape along the basal plane and are randomly oriented in the observation plane. Therefore, characterizing the grain size of such a microstructure might be very intricate but also convenient to compare results from this study with the ones reported by other authors. In this study, polarized light micrograph (Fig. 1c) was analyzed in order to investigate the average grains size according to three different methods based on

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