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Precipitation of γ' phase in δ-precipitated Alloy 718 during deformation at elevated temperatures

S.A. Nalawade, M. Sundararaman*, J.B. Singh, A. Verma, R. Kishore

Structural Metallurgy Section, Mechanical Metallurgy Section, Bhabha Atomic Research Centre, Mumbai 400085, India

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ABSTRACT

Alloy 718 samples aged to precipitate only δ particles (with maximum volume fraction) when tensile deformed to fracture at elevated temperatures revealed precipitation of γ' and γ'' phases. The γ' precipitation was found to precede the γ'' phase precipitation unlike in the case of specimens subjected to standard ageing treatment where both the γ' and the γ'' phases precipitate simultaneously. This sequence is explained on the basis of the relative concentration of Al, Ti and Nb in the matrix of δ precipitated Alloy 718 microstructure. The precipitation sequence was consistent with the Cozar and Pineau's model that predicts such sequences on the basis of (Al + Ti) to Nb atom ratios.

1. Introduction

Precipitation of intermetallic phases in Alloy 718 has been extensively studied and the major phases that precipitate in a face centered cubic (fcc) matrix are metastable γ'' ($D0_{22}$ structure), equilibrium γ' ($L1_2$ structure) and equilibrium δ ($D0_a$ structure) phases [1–4]. Age hardening is mainly brought about by the precipitation of the γ'' phase that are coherent disc shaped particles with {1 0 0} habit plane. Some strengthening is brought about by the precipitation of coherent γ' particles as well [3]. The γ' precipitates form in spherical morphology that transforms to cuboidal upon prolonged ageing. The relative stabilities, the volume fraction and the sequence of precipitation of γ' and γ'' phases are governed by the relative concentration of Al, Ti and Nb in the alloy [4–5]. Cozar and Pineau [5] have shown that the γ' precipitation precedes the γ'' precipitation when the ratio of (Al + Ti) to Nb atoms is greater than about 0.8 and exhibits a compact morphology (where γ'' forms on all six faces of cuboidal shaped γ' particles) when the ratio is between 0.9 and 1 [5]. However, when the γ'' precipitation precedes the γ' formation or both forms simultaneously as in the case of conventional Alloy 718, it becomes difficult to identify their sequence from electron diffraction patterns since all superlattice spots corresponding to the γ' phase are common to the γ'' phase as well. These phases are difficult to identify from their morphology too since both phases exhibit spherical morphology during initial stages of formation [4].

The equilibrium δ phase that forms in the alloy during high temperature processing or prolonged service has plate shaped/blocky morphology [6,7] and its precipitation at grain boundary is reported to have beneficial effect on stress rupture properties [8]. The δ particles have been observed to nucleate from grain boundaries as well as from stacking faults within γ'' particles during ageing at intermediate temperatures [6]. Earlier studies [9,10] have reported that on prolonged ageing/service, the δ precipitation occurs at the expense of metastable γ'' particles while the size and distribution of γ' particles remain more or less unaffected. The application of stress accelerates this process [10]. Similar behaviour has been observed in standard aged Alloy 718 turbine discs under stress at service temperatures close to 650 °C [11].

This paper reports the results of microstructural investigation on the precipitation of γ' and γ'' particles and their sequence during deformation in Alloy 718 samples in which the maximum volume fraction of the δ phase has been precipitated. This work forms a part of detailed research on deformation behaviour of Alloy 718 at elevated temperatures [12]. The observed precipitation sequence has been explained on the basis of relative concentration of solutes participating in the precipitation of γ'' and γ' phases in the matrix of δ precipitated alloy samples.

2. Experimental

The nominal chemical composition of Alloy 718 used in this study was 0.02/C, 52.69/Ni, 18.37/Cr, 18.06/Fe, 6/Nb, 2.91/Mo,

* Corresponding author. Tel.: +91 22 25595368; fax: +91 22 25505151.
E-mail address: msraman@barc.gov.in (M. Sundararaman).

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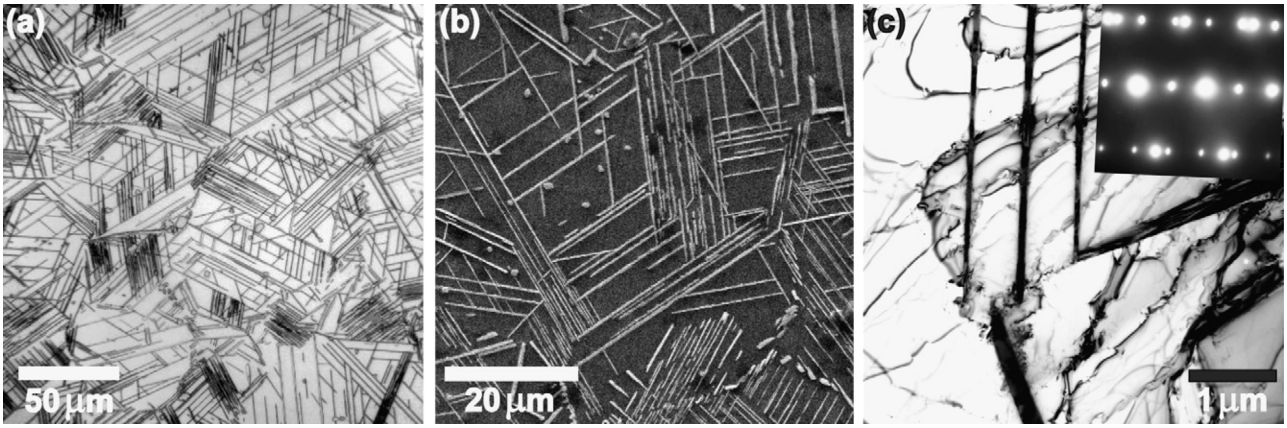

Fig. 1. Representative microstructures of a DELTA heat treated alloy; (a) Optical, (b) SEM and (c) TEM. ⟨1 1 0⟩ zone axis SAD pattern in inset in (c) shows SL spots corresponding to only δ phase.

1/Ti, 0.45/Al, 0.21/Mn and 0.29/Si. Flat tensile samples of gauge dimensions of 1.5 mm × 4.0 mm × 12.6 mm were prepared from cold rolled alloy. The samples sealed in silica capsules under helium atmosphere were solution annealed at 1100 °C for 1 h and water quenched to dissolve all intermetallic phases. The solution treated (ST) samples were further aged at 900 °C for 100 h to precipitate the maximum volume fraction of δ phase (henceforth designated as DELTA specimens). The DELTA specimens were tensile tested up to fracture in air on a screw driven Instron testing machine at a strain rate of 6.5×10^{-5}/s and in the temperature range of 200–700 °C. All samples exhibited ~40% total elongation to failure and the time spent by the sample at the test temperature was of the order of 100 min. The microstructure of DELTA specimens deformed to fracture was examined in optical, scanning electron and transmission electron microscopes. The chemical composition of matrix of specimens subjected to solution and DELTA heat treatments was carried out by energy dispersive analysis (EDS) attached to the scanning electron microscope.

3. Results

3.1. Initial microstructure

The microstructure of ST specimens revealed equiaxed grains (of about 65 μm in size) with single γ phase indicating that this heat treatment was sufficient to dissolve all solutes in solution in the matrix. The details of this microstructure have been reported earlier [13]. The DELTA microstructure revealed uniform and dense distribution of needle shaped δ precipitates in the matrix (Fig. 1). From stereographic analysis, these particles were found to lie on {1 1 1} planes of the matrix in agreement with that reported in the literature [6]. The ⟨1 1 0⟩ zone axis selected area diffraction (SAD) pattern shown as inset in Fig. 1c clearly revealed the presence of superlattice reflections corresponding to only δ phase and not γ′ or γ″ phase. This observation indicated that the precipitation of the γ′ or/and γ″ phase had not occurred during this ageing treatment.

3.2. Deformation microstructure of fractured DELTA samples

TEM examination of specimens from delta samples deformed to fracture at test temperatures below 600 °C revealed high density of dislocations confined within bands in the matrix region between δ particles (Fig. 2a). Dislocation activity was noticed within the δ phase as well. However, precipitation of either of the γ′ or γ″ phases was not observed in the matrix region between δ particles. This was evidenced by the absence of superlattice reflections corresponding to the two phases in the SAD pattern taken from the matrix regions (Fig. 2b). At test temperature of 600 °C, in addition to high density of dislocations generated due to deformation in the matrix (Fig. 3a), the [1 1 2] zone axis SAD pattern, taken from the region between δ particles showed superlattice reflections at specific positions (Fig. 3b). These superlattice reflections could correspond to γ′, γ″ and/or δ phases as depicted in simulated diffraction patterns given in Fig. 3c–e, respectively. Dark field (DF) imaging with

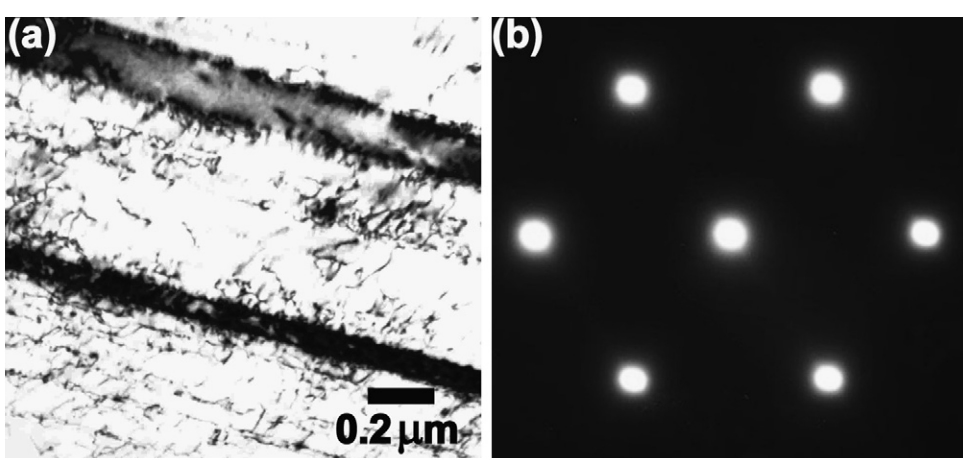

Fig. 2. Deformation microstructure of DELTA specimen tested at 500 °C; (a) BF showing uniform distribution of dislocations in the matrix, (b) SAD pattern from the matrix region. SL spots corresponding to γ′ and γ″ were not seen.