Materials Science & Engineering A 574 (2013) 86–93

Contents lists available at SciVerse ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Fracture behavior of Alloy 625 with different precipitate microstructures

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ARTICLE INFO

Article history:
Received 24 January 2013
Received in revised form
17 February 2013
Accepted 9 March 2013
Available online 16 March 2013

Keywords:
Fracture
Nickel based superalloys
Mechanical characterization
Electron microscopy
Aging
Precipitation

ABSTRACT

To study the effect of various ordered phases and their growth on room temperature tensile properties and fracture behavior of Alloy 625, specimens of this alloy were held isothermally at 813 K, 973 K and 1123 K for 10 h, 100 h and 1200 h, respectively. Specimens held at the aforementioned temperatures were subjected to transmission electron microscopic investigation to characterize $Ni_2(Cr,Mo)$, γ'' and δ ordered phases. Tensile testing of the heat-treated samples revealed the influence of these ordered phases on the tensile properties and fractures. Primary carbides, which were identified semi-qualitatively under electron-probe micro-analyzer (EPMA) have been found responsible for void nucleation resulting in large dimples in Alloy 625 with solid-solution matrix. Early stages of $Ni_2(Cr,Mo)$ and γ'' and their subsequent growth at 813 K, was responsible for increase in strength without reducing ductility considerably. This behavior has been correlated with the reduction in size and depth of the dimples in the fractographs. Transmission electron micrographs showing three variants of γ'' in three orthogonal directions in the alloy matrix have revealed that the morphology of the ordered phase is lens shaped and the precipitate can grow up to ~150 nm. With the growth of γ'' the alloy is seen to fracture in transgranular cleavage manner following ~23% of uniform strain. Influence of plate shaped, long and thick δ phase on the strength and ductility appears similar to that of γ''. δ—precipitation after 1200 h of isothermal holding at 1123 K is also seen to cause faceted appearance of the fracture surface, which is similar to that of γ'' after 1200 h of isothermal holding at 973 K. However, the facets are seen to be consisting of dimples, which are small and uniformly distributed over the surface.

1. Introduction

Alloy 625 is a Ni-base alloy used for high temperature structural applications. Although, the alloy was designed to derive its high temperature strength from solid solution strengthening, it is now known that several ordered phases form during operation at high temperatures [1–5]. It has been reported that prolonged use of the alloy at high temperatures results in an unacceptably low room temperature ductility, thus, jeopardizing its handling at room temperature [6] and possibly increasing the risk of failure [7]. In these reports, Alloy 625 components were subjected to high temperature operations for a long duration having imprecise time-temperature histories. Analysis of such unservice worthy Alloy 625 components has resulted in identifying several microstructural features responsible for variation in strength, which may be critical during service. Although, the effect of aging on the alloy properties is known to be significant, for the want of data from reliable laboratory experiments a systematic correlation in the structure-property area, delineating the role of various precipitates on mechanical properties and fracture of the alloy is yet to be established.

Influence of γ' precipitates on the fracture-surface morphologies of various Ni-base IN738LC and CMSX-4 alloys has been reported earlier [8,9]. It has been observed that the variations in distribution and morphology of this second-phase may result in a wide range of fracture surfaces, such as, dimple-ductile, quasi-cleavage and cleavage types. Characteristics of the fracture surfaces and the crystallographic planes of the facets are seen to depend upon the size of the precipitates and the type of the loading. It has been reported that IN738LC microstructure with fine γ' (70 nm) and with the mixture of fine and medium sizes, of 70 nm and 450 nm, respectively, produce transgranular quasi-cleavage type fracture. The fractographs obtained from the samples tested at room temperature are reported to have facets comprising {100} type crystallographic planes [8]. However, the samples tested at high temperature are reported to also produce {111} type facets [8]. Inability of the fine precipitates to initiate voids has been reported to be responsible for such faceted cleavage type fracture. Microstructure with γ' precipitates of medium size and of coarse size, 450 nm and 700 nm, respectively, are reported to be responsible for void nucleation, which resulted in the dimple type fracture surface. Such dimple-ductile fracture is also seen with coarser carbide precipitates, of 1–2 μ, at the grain boundary which resulted in the micro-crack in the region. However, during room-temperature fatigue, fatigue cracks are reported

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http://dx.doi.org/10.1016/j.msea.2013.03.021

Table 1
Composition of Alloy 625 (wt%).

Ni	Cr	Mo	Nb+Ta	Fe	Co	Al	Ti	Mn	Si	C	P	S
~62	20–23	8–10	3.15–5.15	5.0	1.0	0.4	0.4	0.5	0.5	0.1 max.	0.015 max.	0.015 max.

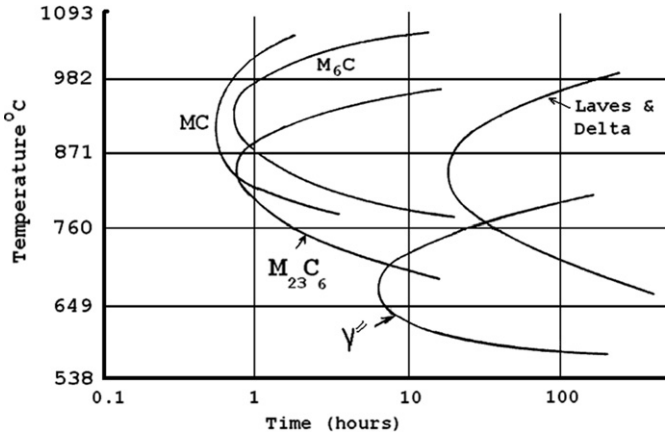

Fig. 1. Time–temperature–transformation diagram of Alloy 625 [1].

to cut through γ′ precipitates and propagate along {111} planes, resulting {111} plane type faceted fracture surface in Ni-base CMSX-4 alloy.

Alloy 625 is essentially a solid solution of molybdenum and niobium in a nickel–chromium-base matrix as per the nominal composition given in Table 1.

The Time–temperature–transformation (TTT) diagram of Alloy 625 is shown in Fig. 1 [1]. It is apparent from the TTT diagram that various types of carbides, such as, MC ('M' rich in Nb, and Ti), $M_{23}C_6$, ('M' rich in Cr), M_6C ('M' rich in Ni, Nb and Mo) form in this alloy and their occurrences depend upon various factors, including, history of solidification, carbon concentration and temperature of holding. Apart from the carbides and Lave's phases, most of which enter the microstructure during solidification process, several other ordered phases, such as, γ′ (Ni_3[Ti,Al]), γ″ (rich in Ni and Nb), δ (Ni_3[Nb,Mo]), Ni_2(Cr,Mo) also precipitate out in Alloy 625 [1]. The TTT diagram, however, does not show Ni_2(Cr,Mo) phase that forms on aging below 873 K and also known to dissolve on heating above this temperature [10].

In the present work, an attempt has been made to correlate the influence of various ordered phases, such as, Ni_2(Cr,Mo), γ″ and δ on the mechanical properties and fracture of the alloy. To benefit from the kinetics of transformation, temperatures, close to the noses of the "C" curves of those ordered phases in the TTT diagram have been chosen for treating the alloy isothermally. E.g. 1123 K has been chosen for the formation of δ, while 973 K has been chosen for having γ″ in the Alloy 625 matrix. However, choice of 813 K, for the formation of Ni_2(Cr,Mo) has been based upon a prudent guess, since, "C" curve corresponding to Ni_2(Cr,Mo) phase is not available in the existing TTT diagram. As stated earlier, too high a temperature, typically above 873 K, is reported to produce a matrix where γ″ dominates, whereas, too low a temperature is likely to slow down the kinetics of transformation, per se. Also, past experience suggests that the influence of Ni_2(Cr,Mo), formed at 813 K, on the microstructure and mechanical properties, is likely to dominate over that of γ″, since, the latter phase is known to be weaker than Ni_2(Cr,Mo), initially [5] and remains dissolvable through shear deformation even after 1200 h of growth [11]. It is also known from the literature that the strength and weldability of the alloy improves just by solutionizing Ni_2(Cr,Mo) selectively at 923 K [6,10].

2. Experimental

Threaded tensile specimens, having 28 mm gage length and 6 mm diameter were machined out from a solution-quenched (SQ) Alloy 625 block, with the gage length oriented along the direction of maximum flow in the wrought working operation. These specimens were held isothermally inside three different resistance-heating furnaces, which were set at 813 K, 973 K and 1123 K. Specimens were taken out of the furnaces after 10 h, 100 h and 1200 h, respectively. Hence, including the SQ material, 10 varieties of samples were tested in the present study.

The tensile specimens were tested using a screw driven INSTRON universal testing machine at a crosshead speed of 0.5 mm/min, giving a nominal strain rate of 3.0×10^{-4} s^{-1}. Load vs. displacement data were recorded in an attached computer, and the output was processed for various flow properties of the material.

Microstructures of SQ samples were investigated using CAMECA SX100 electron probe micro-analyzer (EPMA). Tensile fracture surfaces of SQ and aged samples were viewed using scanning electron microscope Phillips XL30. Transmission electron microscopic study of the samples after 1200 h of isothermal holding at 813 K, 973 K and 1123 K has been carried out using 200 kV JEOL and TITAN transmission electron microscopes to investigate the nature of the second phases.

3. Results

3.1. Tensile properties

Various tensile parameters obtained from SQ and aged samples are summarized in Table 2. Main observations on the trends on tensile properties which could be observed from this table are as follows:

(i) For all the three aging temperatures, strength and ductility values of the alloy samples are seen to vary with the duration of aging, albeit, with different trends. The SQ samples show maximum ductility and near lowest yield strength (YS). Other two specimens, aged at 973 K for 10 h and aged at 1123 K for 10 h, have recorded the second lowest and the lowest YS values, respectively.
(ii) YS of specimens with 10 h of isothermal holding shows decreasing trend with the increase in holding temperature.
(iii) Upon comparing with respect to that of SQ sample rising trends in YS and UTS with the duration of aging are seen for 813 K and 973 K but not for 1123 K aging temperature. In the case of latter temperature, rise in the YS is seen to be slow and UTS is seen to fall, till 100 h of holding.
(iv) Trends in the variation in ductility are seen to be different from those of strengths. Ductility is seen to fall with the duration of holding for all isothermal temperatures except for 813 K, where ductility is seen to rise after 100 h, albeit, maintaining the overall trend in ductility falling.

3.2. Fractographs

The fractograph in Fig. 2 is from the SQ sample that shows large dimples (marked with the arrows). Particles responsible for the