Experimental Report

26/03/2014

Proposal: **1-01-127** **Council:** 4/2012

Title: Omega phase particles in LCB titanium alloy

This proposal is a new proposal

Researh Area: Materials

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Samples: Ti-4.5Fe-6.8Mo-1.5Al (wt. %) i.e. Ti 89.9%, Mo 3.4%, Fe 4%, Al 2.7% (at. %)

Instrument	Req. Days	All. Days	From	To
D11	3	2	03/04/2013	05/04/2013

Abstract:

Titanium alloys are generally two phase containing the hcp alpha – phase and bcc beta – phase. Also unstable omega phase is formed during aging of metastable beta-Ti alloys. It has been shown that volume fraction, distribution and size of omega particles are decisive for the mechanical properties. Ageing kinetics of omega phase formation is not fully understood. Extensive set of 30 samples has been prepared for ex-situ investigation. Three aging temperatures were utilized and ageing times of 15 minutes to 1024 hrs on logarithmic scale were employed. Different chemistry (Mo and Fe content) of omega particles and beta phase matrix allow the distinction of those particles in specimen. In-situ investigation (heating up to 900°C with rate of 1 - 5 °C/min) will give more insight to previously documented consequent phase transitions with growing temperature (beta to omega, omega to alpha, beta to alpha and finally alpha to beta). In-situ experiment is therefore also proposed. Investigation utilizing small angle neutron scattering at ILL will bring detailed information about omega phase particles size, shape and volume fraction in Ti LCB alloy that cannot be acquired by any other method.

1 Introduction

Metastable β titanium alloys are such alloys, in which martensitic $\alpha \rightarrow \beta$ transition is suppressed upon quenching by addition of sufficient amount of β stabilizing elements. In a certain composition range, nano-sized particles of a metastable ω phase form in these metastable β titanium alloys. ω phase particles have a significant impact on mechanical properties of the material as well as on following phase transformations occurring during ageing. As ω phase has hexagonal structure which is coherent with body-centered cubic lattice of parent β phase, its presence in the alloy leads to higher hardness but also to increased brittleness. Furthermore, ω phase particles serve as nucleation sites for thermodynamically stable α phase. This ω assisted nucleation of α precipitates results in very fine, homogeneously dispersed α lath microstructure which in consequence leads to desirable mechanical properties of the material - high strength without any significant loss of ductility.

The aim of this experiment was to observe the evolution and growth of ω phase particles in metastable titanium alloy TIMETAL LCB. Moreover, the influence of ageing time and temperature on the size, shape and spatial ordering of ω and α precipitates was observed.

2 Experimental

In order to observe scattered neutron intensity from one grain in a specific orientation, single crystals of TIMETAL LCB were grown in an optical floating zone furnace. The growth was done in a protective Ar atmosphere with pressure 2.5 bar and flow of 0.25 l/min. The precursor had a cylindrical shape with diameter of 8 mm an length of approximately 100 mm. After the growth process, the orientation of the ingots was determined by Laue method. The single crystals were then solution treated at 860 ℃ for 4 h in an evacuated quartz tube and water quenched in order to ensure retention and homogenization of β phase.

Three series of samples were cut from the solution treated single crystals in orientations $\langle 100 \rangle_\beta$, $\langle 110 \rangle_\beta$ and $\langle 111 \rangle_\beta$ perpendicular to the sample surface (i.e. parallel to the beam in the experimental setup). The samples were then aged at different ageing temperatures and times in range of ω phase growth as well as α precipitation according to table below.

T \ t	5 min	2 h	4 h	8 h	16 h	32 h	64 h
300 ℃				X			X
335 ℃			X			X	
370 ℃		X			X		
490 ℃	X						
510 ℃	X						

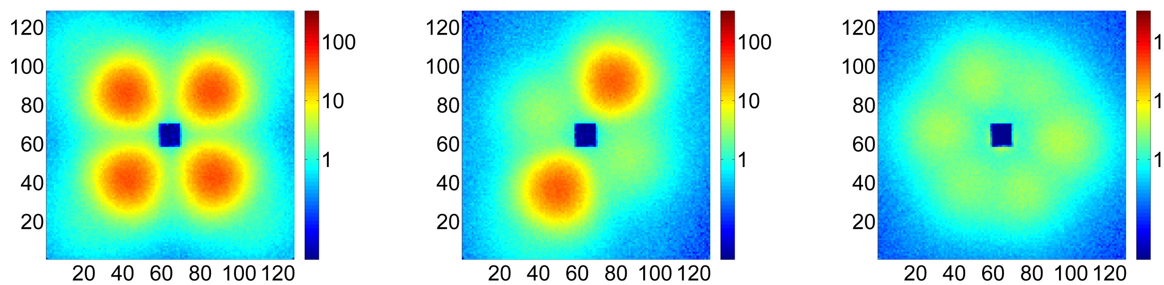
For the experiment, the samples were ground to a thickness of approximately 1 mm and polished utilizing vibratory polisher.

Three sample–detector distances were used in order to observe a wider range of q-vector: 1.2 m, 2.5 m and 8 m.

Furthermore, in-situ experiments during heating were done on solution treated samples.

3 Results

The measurement of small-angle neutron scattering on aged samples gave very satisfactory results - clear dependence of ω particle size on ageing temperature and time was observed. Furthermore, multiple distinct scattering maxima were observed. The symmetry of the maxima was dependent on the orientation of the sample (i.e. whether the $\langle 100 \rangle_\beta$, $\langle 110 \rangle_\beta$ or $\langle 111 \rangle_\beta$ direction was parallel to the primary beam), which implies spatial ordering of ω particles. An example of measured scattering patterns and their dependence on the orientation of the sample is given in Fig. 1.



a) b) c)

Figure 1: Small-angle neutron scattering patterns measured in a) $\langle 100 \rangle_\beta$, b) $\langle 110 \rangle_\beta$ and c) $\langle 111 \rangle_\beta$ direction parallel to the beam

According to an independent Laue diffraction measurement, we determined that the main scattering maxima are in $[100]_\beta$ directions. That means that the ω particles are ordered in a cubic lattice with edges along $[100]_\beta$.

Two in-situ heating runs were done with solution treated samples. The chamber of the furnace was first evacuated to a high vacuum (up to 10^{-6} Pa), then the temperature was raised and held at 370 ℃ (formation of ω phase particles) and at 490 ℃ (precipitation of α phase). Growth of ω particles evidenced by circular maxima in the first case and growth of α laths evidenced by streaks in scattering pattern in the second run was observed. However, the early stages of ω and α particles formation could not be observed due to long time of data acquisition - these processes are much quicker than typical signal acquisition time (approx. 600 s).

4 Conclusion

The small-angle neutron scattering experiment at ILL yielded very valuable data, which in combination with results of other experimental techniques (e.g. small-angle X-ray scattering, X-ray diffraction etc.) will help us understand the process of formation and growth of ω and α particles in metastable β titanium alloys.