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INFLUENCE OF AGEING ON THE INTERNAL FRICTION OF Nb-Ti-N ALLOYS

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Abstract. - We have studied the influence of ageing on the internal friction of various Nb-4.6Ti-N alloys at 1Hz, annealed for 24 hours at 400, 600, and 800°C. This study shows the influence of temperature, beginning at 400°C, on the measurements of internal friction and allows a more complete description of the structure and behaviour of Nb-Ti-N alloys.

Introduction. - The study of the ageing of an alloy can be performed using various methods. The internal friction technique allows the ageing process to be followed with great accuracy and it yields a lot of information, especially on the quenching ability of solid solutions. In previous publications (1,2), we presented results concerning the study of internal friction, structure and mechanical properties of Nb-4.6%at.Ti-N quenched alloys : an instability of the internal friction measurements beyond 400°C, and a modification of the internal friction spectra at the time of a second determination at increasing temperature were observed. In order to specify the origin of these modifications, we performed new studies on those alloys after ageing treatments at different temperatures.

Material and experimental procedures. - The material used is a Nb-4.6%at.Ti alloy, already described in a previous work (1). Interstitial contents of these alloys are : 0.65, 0.85, 1.3, 2.0 and 2.6 % at.N.

Specimens of these alloys (except the 2.6 % at.N alloy), initially quenched from 1750°C, were annealed for 24 hours, respectively at 400, 600, and 800°C, in vacuum sealed tubes (10^{-4} Pa), and then quenched in cold water.

The Nb-4.6Ti-2.6N alloy was aged in situ in the pendulum. This ageing treatment consisted in successive annealings run on the same specimen : the internal friction measurements were performed at increasing and decreasing temperatures. The upper temperature of each successive annealing was increased by 50 degrees and the sample was maintained at this temperature for one hour.

Internal friction measurements were carried out in an inverted vacuum ($\sim 10^{-4}$ Pa) torsion pendulum apparatus operating at a frequency of about 1Hz. The length of the specimen was 45 mm and it was heated from room temperature up

to 600°C at a constant rate of 1 degree per minute.

The complex damping spectra were resolved into Debye peaks by a computer method using successive substractions.

The internal friction study of those alloys was completed by microscopic examination, lattice parameter determination and tensile tests.

Results. - The microscopic examination revealed a beginning of precipitation at the grain boundaries from the specimen containing 2 % at.N aged at 800°C. The X-ray determination of the lattice parameter showed a small increase for the samples aged at 800°C, probably due to an oxygen contamination during annealing.

The values corresponding to the tensile tests are reported in table I.

		Quenched at 1750°C	Aged at 400°C	Aged at 600°C	Aged at 800°C
0.65 % at.N	R MPa	540	550	608	~ 600
	E 0.2% MPa	490	490	590	-
	A%	10	10	6	0.5
0.85 % at.N	R	750	680	640	~ 500
	E	700	590	565	-
	A	12	12	10	0.4
1.3 % at.N	R	840	630	520	~ 400
	E	760	580	465	-
	A	8	7	4	0.2

TABLE I

Furthermore, the examination of the broken surface shows a ductile rupture with an important reduction in area, for the samples with an interstitial N content of 0.65 % at. and aged at 400 and 600°C, and a brittle, intergranular fracture, for the other samples.

The results of the internal friction measurements are reported on figure 1 for the dynamic ageing determination, and on figure 2 for the ageing study at 400, 600 and 800°C.

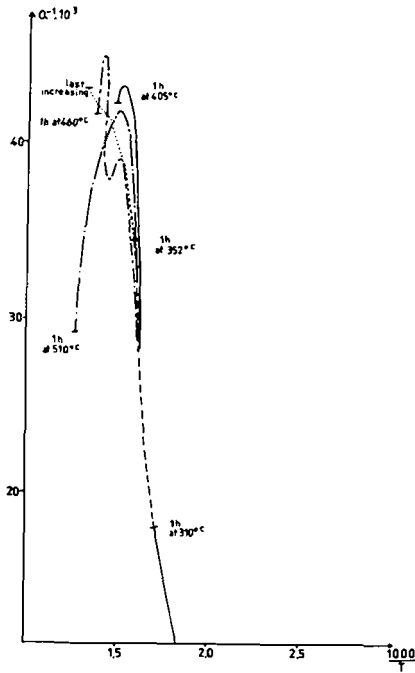


Figure 1

Figure 1 shows that, for an annealing temperature higher than 400°C , an important instability of the solid solutions is revealed by the non-reproducibility of the internal friction measurements. These deviations are due either to a new distribution of nitrogen atoms in the matrix or to a precipitation phenomenon which increases with temperature.

The curves shown in figure 2 have been decomposed in several elementary peaks (Table II). The same peaks were observed either on the aged samples or on the quenched samples or on the quenched ones. However, a new peak appeared at 560°C for the specimens aged at 600 or 800°C . Furthermore, the samples containing 1.3 and 2.0 % at.N show a supplementary peak at 385°C .

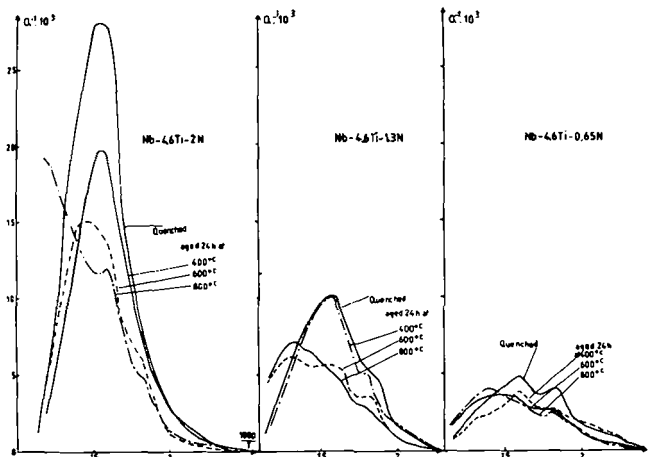


Figure 2

% at.N	t°C of quenching	280°C	345°C	385°C	410°C	485°C	560°C
0,65 %	quenched	3.7	3.9		3	2.2	
	400°C	2.3	3.4		3	1.4	
	600°C	2.2	2.9		2.8	2.7	1.6
	800°C	2.2	2.8		3	2.7	1.8
0,85 %	quenched	5.4	5.3		4.2	3	
	400°C	4.7	5		3.8	2.4	
	600°C	3.7	4		3.6	1.9	4
	800°C	2.3	3.7		3.3	3.8	5
1,3 %	quenched	5.3	8.2	8.5	8.2	5.1	
	400°C	3.8	5.3	7.7	7.4	4.4	
	600°C	2.7	4.3	4.3	6	6	5.8
	800°C	2.7	3.6	4.3	4.3	4.7	6.8
2 %	quenched	8	22	16	18	9.2	
	400°C	6.1	14.7	14.5	11.1	5.2	
	600°C	5.8	11.6	11.6	11.2	5	5
	800°C	5.8	11.8	9.8	14.2	8	17.6

TABLE II

Discussion. - These results allow us to make the following remarks :

- Firstly, about the mechanical properties, an ageing at 400°C involves few modifications for the samples with small interstitial content (< 0.85 % at.N). But after an annealing at 600 and 800°C a hardening with a decrease in ductility was observed. This was probably due to a microprecipitation of the nitride TiN. For the most loaded alloys (≥ 0.85 % at.N), a precipitation at the grain boundaries was noticed after annealing at 400°C (or more) and this diminution of the interstitial content involved a decrease in the mechanical characteristics.

- Secondly, about the internal friction measurements : the variation of the different elementary peaks versus nitrogen content is reported on **figure 3** for various annealing and quenching treatments :

. the intensity of the first four peaks decreases when the ageing temperature increases and the extrapolation for an interstitial content being equal to zero shows a convergence of all the curves towards zero. This fact strengthens the hypothesis that those peaks are caused by the reorientation of nitrogen atoms in solid solutions.

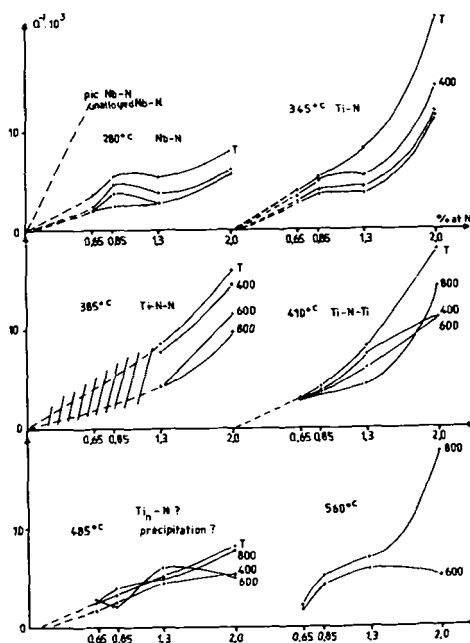
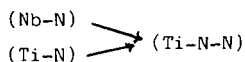


Figure 3

. the peaks at 280 and 345°C present an anomaly in the domain 0.65–1.3 % at.N. which could be produced by an abnormal behaviour of nitrogen in this interval, followed by a redistribution of the nitrogen atoms, at a higher concentration, and in another kind of interstitial sites.



. the origin of the peak at 560°C is uncertain. We think that it can be due to a relaxation phenomenon of nitrogen atoms near precipitates and would have some analogy with a cold-worked peak (3). The irregular variation of the 485°C peak does not allow a conclusion to be made about its origin.

Conclusion. - These results show the importance of internal friction in order to get information about the ageing of alloys. Together with lattice parameter and mechanical determinations, for example, this method allowed a better knowledge of the behaviour of interstitial nitrogen in a Nb-Ti alloy.

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