FTD-MT-24-14

FOREIGN TECHNOLOGY DIVISION

EFFECT OF NITRIDING ON THE MECHANICAL PROPERTIES OF NIOBIUM

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12

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Foreign Technology Division Air Force Systems Command U. S. Air Force	2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED 2b. GROUP

3. REPORT TITLE
EFFECT OF NITRIDING ON THE MECHANICAL PROPERTIES OF NIOBIUM

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)
Translation

5. AUTHOR(S) (First name, middle initial, last name)
S. S. Kiparisov, Yu. V. Levinskiy, and Yu. D. Strogonov

6. REPORT DATE 1970	7a. TOTAL NO. OF PAGES 7	7b. NO. OF REFS 9

8a. CONTRACT OR GRANT NO. b. PROJECT NO. 60107/T68-01-02 c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) FTD-MT-24-1431-71 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. DISTRIBUTION STATEMENT
Approved for public release; distribution unlimited.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Foreign Technology Division Wright-Patterson AFB, Ohio

13. ABSTRACT

The effects of nitriding on the mechanical properties of Niobium (short-term tensile strength, plasticity, hardness) are discussed. An optimal nitriding regime is found for the surface-hardening of Niobium.

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DD FORM 1 NOV 65 1473

UNCLASSIFIED

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Niobium						
Nitrogen						
Plasticity						
Elongation						
High Temperature						
Nitriding Process						
Mechanical Properties						

FTD-MT- 24-1431-71

EDITED MACHINE TRANSLATION

FTD-MT-24-1431-71

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English pages: 7

Source: Izvestiya Vysshikh Uchebnykh.
Zavedeniy. Tsvetnaya Metallurgiya (News
of Institutions of Higher Learning.
Nonferrous Metallurgy), No. 4, 1970,
pp. 125-128.

Requester: FTD/PDTI

Translated by: M. Olaechea

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	PREPARED BY: TRANSLATION DIVISION FOREIGN TECHNOLOGY DIVISION WP-AFB, OHIO.

FTD-MT- 24-1431-71 **Date** 4 May **19** 72

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О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

EFFECT OF NITRIDING ON THE MECHANICAL PROPERTIES OF NIOBIUM

S. S. Kiparisov, Yu. V. Levinskiy,
and Yu. D. Strogonov

Niobium has a relatively high strength and plasticity and excellent chemical resistance. Yet due to the low hardness of the pure metal, items produced from it have poor wear resistance. In this connection processes for surface hardening of niobium products and, in particular, a simple and effective nitriding method in a nitrogen atmosphere are subjects of great interest.

Several works [1-6] have been dedicated to a study of nitriding, yet in most of these attention is given primarily to studying the kinetics and mechanism of the process.

In this work we have studied the effect of nitriding on the mechanical properties of niobium: short-term tensile strength, plasticity, and hardness.

Niobium of an electron-beam melt, rolled without heating into sheets 0.7, 1.6, and 2.0 mm thick with a 97, 94, and 92% degree of deformation, respectively, was used as the raw material. From these sheets specimens were cut for testing tensile strength (total length 50 mm, width of the end 3 mm).

in the original cold-worked state σ_B = 60-63 kg/mm², δ = 5 - 13%, and HV = 210-220 kg/mm². Annealing was conducted in a vacuum of $1 \cdot 10^{-5}$ mm Hg, for one hour at 1100, 1200, and 1400° up to the reduction of σ_B to 32, 30, and 21 kg/mm², an increase of δ up to 23, 22, and 20%, and a reduction in hardness to 110, 109, and 94 HV, respectively.

Annealed as well as cold-worked specimens were subjected to nitriding. After nitriding at all test regimes, no difference, either in structure or in the properties of these two specimen batches, was revealed. Evidently, recrystallization annealing occurred as the specimens were being heated up to the nitriding temperature. This theory is confirmed by the data on the temperature of 940-1050° at the beginning of the niobium recrystallization [7].

Nitriding was performed in an atmosphere of pure nitrogen containing less than 0.005% oxygen. The specimens were saturated by nitrogen at 1100-1500° with a holding time of from 15 min to 6 h. For the nitriding the specimens were suspended on a molybdenum wire in the working area of a vacuum furnace with a tungsten heater. Prior to heating a vacuum of $1 \cdot 10^{-3}$ mm Hg was created in the furnace. After the interior of the furnace had been flushed three times with nitrogen, a nitrogen pressure equal to 1 atm was established, and remained constant throughout the entire experiment. The temperature on the surface of the specimens was measured by means of an optic pyrometer with an accuracy up to 10-20°.

Over the entire studied temperature interval during nitriding two processes took place simultaneously, but at different rates and with different effects on specimen hardness. These were the saturation of niobium by nitrogen, which increased hardness, and the growth of brittle nitride layers, which decreased the overall strength of the specimens.

A weight analysis provides a qualitative picture of the relative participation of these processes in changing specimen strength. The ultimate solubility of nitrogen in niobium at saturation temperatures according to W[illegible]k [6] is equal to 0.04 wt.% N. According to data on the quantity of absorbed nitrogen we can determine the time during which the process of saturation of the metal by the nitrogen is terminated and all the absorbed nitrogen spent in the growth of nitride layers (table).

Time (hour) necessary for the formation of a maximally saturated solid solution of nitrogen in niobium.

t, °C	$\delta = 0{,}7$ мм	$\delta = 1{,}6$ мм	$\delta = 2{,}0$ мм
1000	≈6	—	—
1100	3	>6	—
1200	0,5	1	1
1300	≈0,1	0,25	0,25

The nitrided specimens were tested for tensile strength at room temperature. With an increase in nitriding time at 1100° the strength of the specimens rises, which is explained by the concentration of nitrogen in the solid solution (Fig. 1). The thinner the specimen, the more rapid will be its saturation and the greater the increase in its strength. Evidently, σ_B on the order of 70 kg/mm^2 corresponds to the ultimate saturation solid solution of N in Nb. The films of the brittle nitride phases on the surface of the specimens at 1100° are still very small and do not play a significant role in the overall hardness of the specimens.

At 1200° (see Fig. 1b) nitrogen saturation of the solid solution occurs more rapidly. Specimens 0.7 mm thick have a maximum hardness after only 1-[illegible] h of nitriding, and in this case the formation of nitride layers has a significant influence on strength. Since the strength of a solid solution after ultimate nitrogen saturation remains constant, while the nitride layers continue to grow, the relative contribution of these layers to the overall strength of the specimens increases with an increase in holding. Actually, the strength of specimens 0.7 mm thick declines somewhat with an increase in nitriding from [illegible] to 6 h.

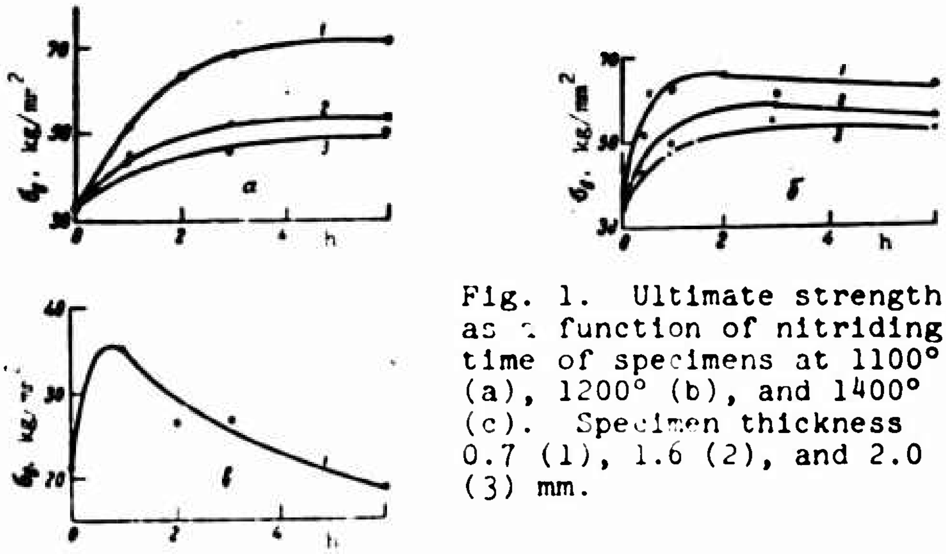

Fig. 1. Ultimate strength as a function of nitriding time of specimens at 1100° (a), 1200° (b), and 1400° (c). Specimen thickness 0.7 (1), 1.6 (2), and 2.0 (3) mm.

At 1300° saturation of the solid solution by nitrogen is completed after only 15 min. Then the formation of nitride layers begins to affect the change in strength. The greater the relative thickness of these layers, i.e., the thinner the specimens, the greater will be the decline in strength with an increase in time.

We get a similar picture at 1400° (see Fig. 1c), where strength after nitriding for 6 h becomes even less than that of specimens in an annealed state.

At all nitriding temperatures the sharp decline in plasticity is quite noticeable. At 1100° there is a two-fold decrease in relative elongation during the first two hours of nitriding. A further increase in the nitriding time does not result in a substantial decrease in plasticity. Evidently, δ = 10-14% corresponds to the maximal saturated solid solution of N in Nb. It is interesting to note that the relative elongation of a maximally saturated solid solution of oxygen in niobium has about the same value (10%) [8].

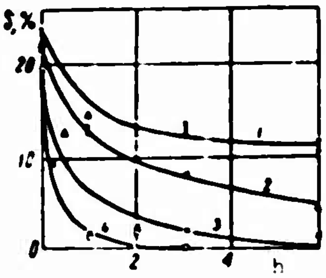

Fig. 2. Relative elongation of specimens 0.7 mm thick as a function of nitriding time at 1100° (1), 1200° (2), 1300° (3), and 1400° (4).

After nitriding at 1200 and 1300° plasticity declines more abruptly than at 1100°. The constant decrease in plasticity and its low values after prolonged nitriding at 1300° indicate that the reduction in strength after prolonged nitriding at 1300° is not caused by the recrystallization processes, which occur under these conditions along with nitrogen saturation, but by the growth in brittle nitride layers (Fig. 2). At 1400° after 1 h of nitriding we observe a virtual brittle fracture of the specimen. The surface hardness values for specimens of different thicknesses, subjected to nitriding under identical conditions, differ little. Thus, Fig. 3 gives the average values for specimens of different thicknesses.

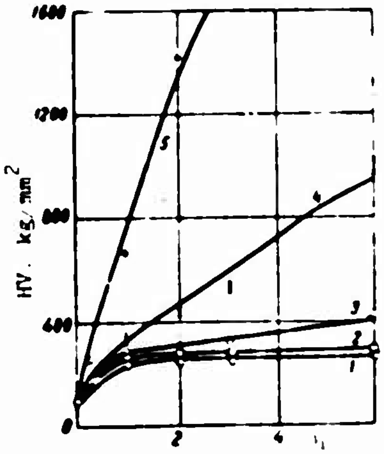

Fig. 3. Time change in surface hardness with nitriding at 1100° (1), 1200° (2), 1300° (3), and 1400° (4).

At nitriding temperatures of 1100-1300° the surface hardness of specimens remains for all temperatures equal to 250-300 kg/mm^2. These values, according to work [9], correspond to a solid saturation solution of N in Nb. A 1400 and 1500° surface hardness rises sharply, and in 2 h of nitriding at 1500° reaches 1500 kg/mm^2. Such high hardness values correspond to the hardness of the nitride layers. In order to measure hardness at a certain distance from the surface of the specimen, a layer 100 μm thick was removed. At this depth hardenss, in contrast to surface hardness, is less dependent on a change in temperature and nitriding time, and within the entire studied temperature range fluctuates within a 200-250 HV range (Fig. 4).

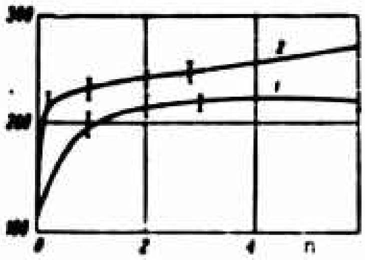

Fig. 4. Change in hardness at a depth of 100 μm from surface during nitriding at 1100° (1) and 1300° (2).

Thus, the nitriding process can be successfully applied in the surface hardening of niobium. The optimal regime in this case is nitriding at 1100° for 2-3 h. After such processing a 230-240 HV surface hardness is obtained, and 210-220 at a distance of 100 μm from the surface. A temperature increase in nitriding is desirable in connection with the great plasticity loss, and for strength loss at high temperatures.

Nitriding can be recommended not only to increase surface hardness, but (in the case of small items) to increase overall strength. The ultimate tensile strength of nitrided specimens is on the order of the ultimate strength of maximally cold-worked niobium - 65-70 kg/mm^2. However, nitrided specimens have a great advantage over cold-worked. While the strength resulting from mechanical working declines sharply with an increase in temperature, strength resulting from the nitrogen saturation of niobium retains its high values even after annealing at elevated temperatures. Thus, annealing of maximally cold-worked specimens at 1100° for 1 h results in a decrease in σ_B from 63 to 32 kg/mm^2; annealing in a vacuum (≈1·10^{-5} mm Hg) at 1200° for 1 h of specimens which have undergone nitriding at 1200° for 1 h resulted in a decrease in σ_B from 62-63 to 52 kg/mm^2.

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Received
14 March 1969