# CHARACTERISTICS OF NIOBIUM-ALUMINUM MASTER ALLOY

# FOR PRODUCTION OF ADVANCED TITANIUM ALLOYS

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Abstract

As a master alloy for niobium bearing titanium aluminides and other advanced titanium alloys, the aluminothermic reduction (ATR) of high-purity niobium oxide to nominal 70Nb-30Al has been produced in the largest single quantity heat (1500 lb.) for consistent quality. The crushed product contained very low dissolved gas content and residual elements. An in-depth analysis and the effect of plasma arc remelting (PAR) of the ATR product was made via optical, SEM, EDS, and X-ray diffraction. The microstructure was dendritic with approximately 67 pct. $NbAl_3$ as the matrix phase and 33 pct. $Nb_2Al$ as the second phase per x-ray diffraction, SEM and EDS. Microhardness measurements revealed that the $Nb_2Al$ was harder than the $NbAl_3$ phase, (696 vs. 557 VHN). PAR of the ATR product resulted in a much finer structure. The cleanliness of both products was very good. Only a small number of inclusions were observed which were cuboidal in shape and EDS analysis revealed that they were rich in Al and Mg, indicating the spinel $MgAl_2O_4$. A demonstration VAR ingot of Ti-24Al-11Nb produced from a compacted electrode of ATR 70Nb-30Al particles and Ti sponge was split longitudinally through the center and it contained no undissolved particles of this master alloy.

## Introduction

The production of niobium bearing titanium aliminides,[1] such as 2411 with 20 wt. pct. Nb, and the Beta 21S alloy,[2] with 3 wt. pct. Nb, should require the addition of consistent quality and higher purity Nb-Al master alloy for blending with high grade Ti sponge for the vacuum arc remelting (VAR) operations. Thus, the degree of cleanliness on hard alpha and inclusions desired by end-users via electron beam cold hearth remelting (EBCHR) and plasma arc remelting (PAR) can be obtained. This paper describes the aluminothermic reduction (ATR) of high purity niobium oxide to nominal 70Nb-30Al in the largest single quantity heats ever produced (1500 lb. at a time) so that a consistent quality level is produced, and it presents an in-depth microstructural analysis of this ATR product and the effect of PAR of the ATR product.

In order to eliminate atmospheric pollution and minimize the impact of cost, CBMM[3] has now installed a new semi-continuous autothermic reduction process for producing ferroniobium in a stationary covered reactor that

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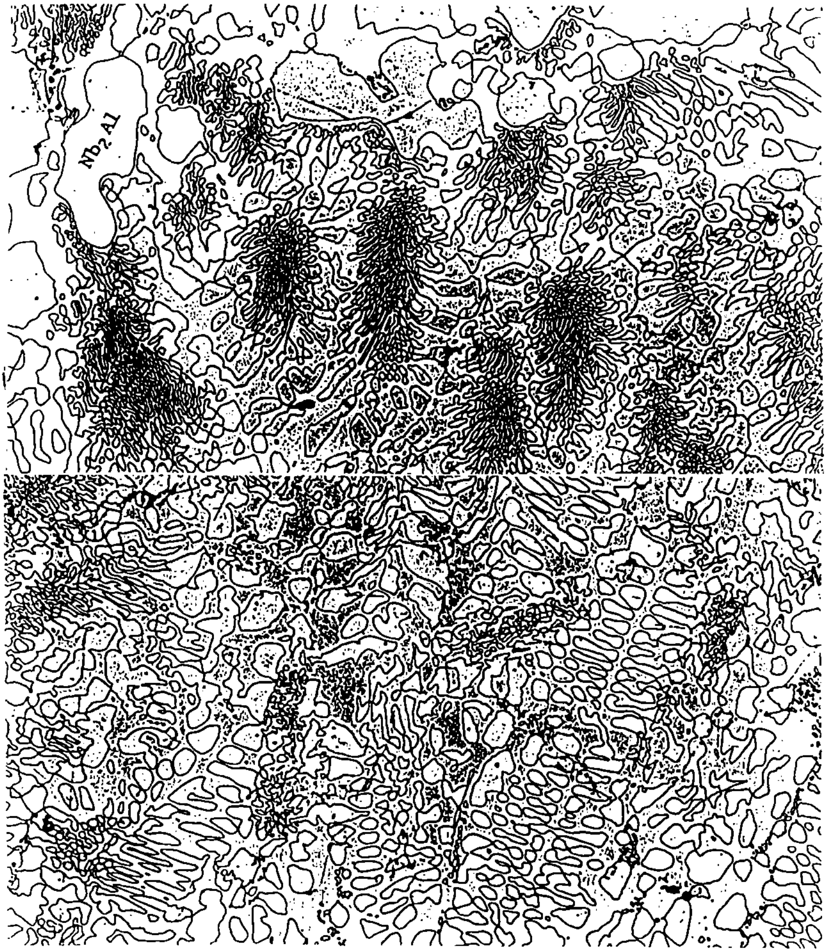


Figure 1. Optical microstructure of ATR 70Nb-30Al product at X200.

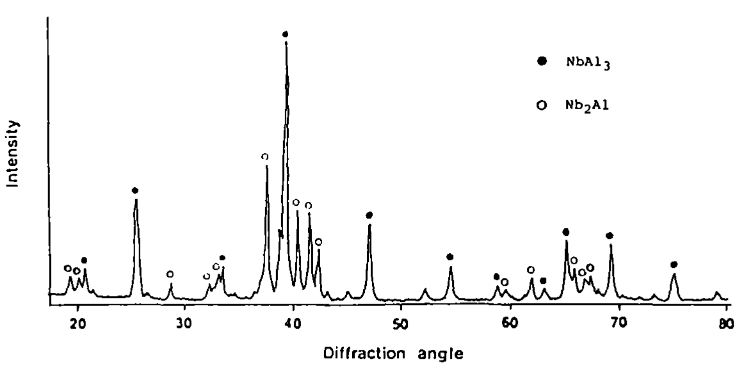


Figure 2. X-ray diffraction patterns of $NbAl_3$ and $Nb_2Al$ phase in ATR 70Nb-30Al.

resembles a normal electric arc furnace. However, the standard process for making ferroniobium from pyrochlore concentrates is a batch process based on aluminothermic reduction (ATR).[4] The CBMM Araxá facility is the largest in the world in terms of reactor size, quantity of Al powder used and tons of alloy produced by each of six daily reactions (11 tons). ATR Nb-4 to 6 pct. Al feed stock for VAR-EB furnace operations is produced in a similar way and the production of 70Nb-30Al is an extension of this practice. The reduction is accomplished in a smaller reactor and the quality of this ATR product, in terms of interstitials, is dependent upon the Al content; the higher the Al the lower the oxygen content.

## Material and Procedure

The crushed ATR product analyzed 69.6 pct. Nb-29.6 pct Al and contained very low dissolved gas content of 45 ppm $O_2$, 55 ppm $N_2$, 10 ppm $H_2$, with the other significant elements being <10 ppm S, <40 ppm P, 0.01 pct. Si, 0.01 pct. C and 0.15 pct. Fe. The overall microstructure after ATR and then after PAR was examined via optical and scanning electron microscopy (SEM). Energy

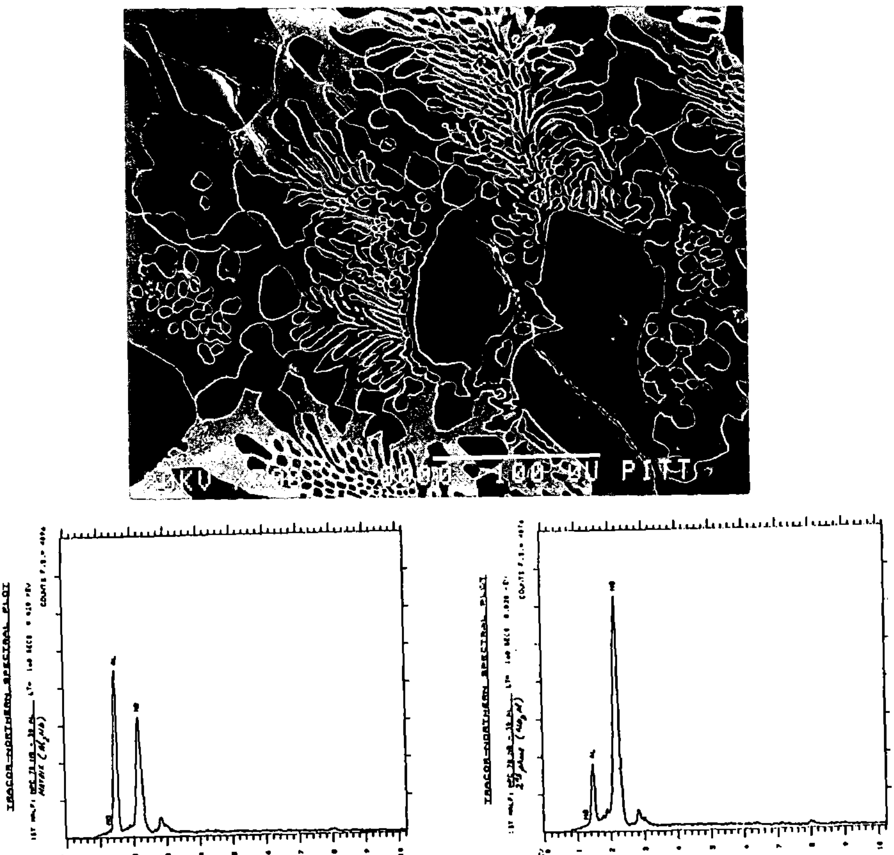

Figure 3. SEM of ATR 70Nb-30Al at X300 and EDS spectral plots of $NbAl_3$ and $Nb_2Al$ phases.

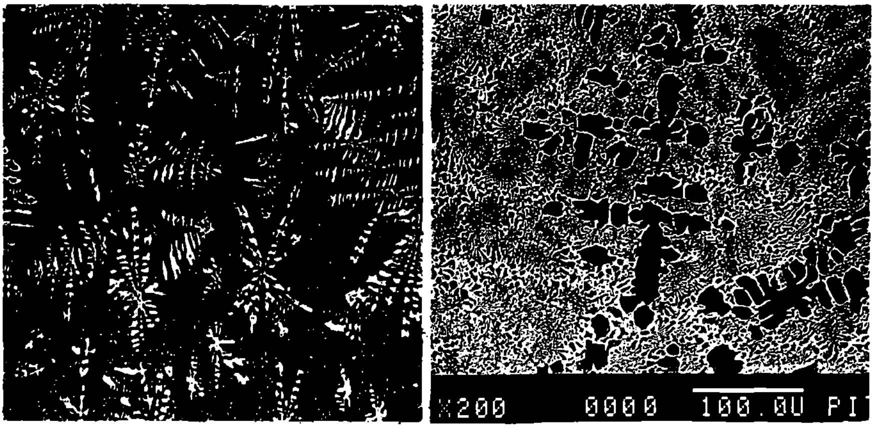


Figure 4. Optical microstructure of ATR 70Nb-30Al after PAR at X150 and SEM at X200.

dispersion spectrometry (EDS) and x-ray diffraction analysis were used to determine the chemical and phase(s) compositions of the matrix and second phase constituents. In addition, the microhardness and volume fraction of the individual microstructural components were determined.

## Results

The overall microstructure of ATR 70Nb-30Al is shown in Figure 1. The structure can be described as dendritic and composed of the $NbAl_3$ matrix and interspersed $Nb_2Al$ phase which appeared in a different tone in the original color photomicrographs at 200 and 600x magnification. X-ray diffraction analysis revealed that the dendritic structure was composed of two phases; $NbAl_3$ and $Nb_2Al$, as seen in the x-ray diffraction pattern of Figure 2. Also, SEM and EDS examination confirmed that the matrix is $NbAl_3$ and the second phase is $Nb_2Al$, per Figure 3. The percentage of each microstructural component by Quantomet is approximately 67 pct. $NbAl_3$ and 33 pct. $Nb_2Al$, with $\pm$ 4.2 experimental error. Microhardness measurements indicated that the second phase $Nb_2Al$ is harder than the matrix $NbAl_3$ phase, the respective values being 696 vs. 557 VHN.

The purpose in the PAR processing of a portion of the ATR product was to ascertain if the dendritic structure could be refined by this secondary operation. By combining superheat to eliminate low-density inclusions and melting on a copper hearth to remove high-density inclusions, plasma yields a very clean fine-grained product, and it maintains the chemical composition of the alloy.[5] As seen in Figure 4, the dendritic structure of the ATR product was refined by PAR. In general, the cleanliness of both the ATR and PAR product was very good, only a small number of inclusions were observed. The chemical composition and morphology of the inclusions in the ATR product are shown in Figure 5. The inclusions are cuboidal in shape and EDS analysis revealed that they are rich in Al and Mg, maybe forming the spinel $MgAl_2O_4$. Inclusions and gas content would be inherent in the quality of the niobium oxide, the ATR practice and the amount of Al in the ATR product. As an example, an ATR 90Nb-10Al contains a much higher dissolved gas content of 270 ppm $N_2$ and 1700 ppm $O_2$ which could contribute hard alpha and inclusions in the remelting operations that produce advanced Ti alloys.

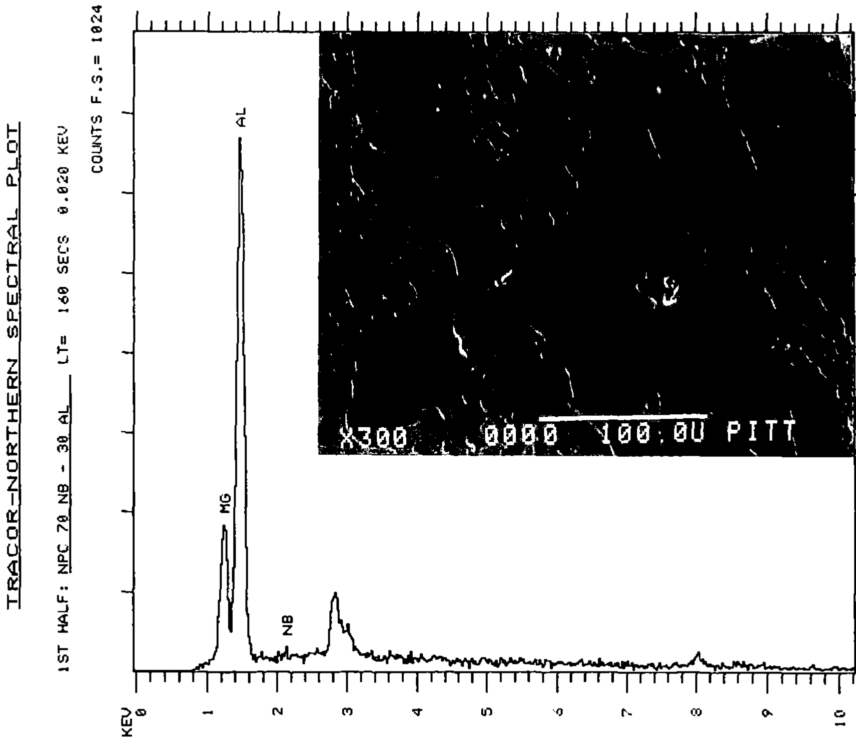


Figure 5. SEM of ATR 70Nb-30Al cuboidal inclusions at X300 and EDS spectral plot revealing Al and Mg content.

The ATR 70Nb-30Al product was ball-milled, without difficulty, to -140+325 mesh powder and is being used as a master alloy addition in the development of Ti-Nb and Nb-Ti alloys. To qualify for Nb-bearing titanium aluminides, a demonstration ingot of nominal 4 in. diameter x 6 in. length of 2411 alloy was cast by VAR of a consumable electrode consisting of the proper particle size relative to the Ti sponge. A macroetch of the longitudinal slice through the center of the ingot is depicted in Figure 6. Any undissolved master alloy particles would act like high-density inclusions or hard alpha particles. In the preparation of this ground/polished surface, they would stand-up as tiny ridges or rough spots felt by hand. Also, the operator did not hear any "clicks" during the surface preparation which would occur if they were present. The macroetch did not reveal any such undissolved particles either visually or under binoculars. It shows the typical elongated grains originating from the starting stub (bottom) and from the water-cooled Cu container, plus the crystallization of smaller, eqiuaxed grains in the center, upper-half of the VAR ingot.

References

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Figure 6. Macroetch of longitudinal, center section of 4 in. diam. by 6.25 in. long demonstration ingot of Ti-14Al-21Nb produced by VAR of compacted electrode of Ti-sponge and 70Nb-30Al master alloy. Composition is Ti-16.1Al-20.4Nb-0.15Fe-0.135$O_2$-0.005$N_2$-0.013C. Actual size.


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