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# Effect of heat input on microstructure and strength of welds in tantalum and niobium alloys

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## Abstract

Welding of niobium and tantalum materials is currently conducted in vacuum or inert gas chambers, which limits the size of the equipment to be produced. The objective of this study is to investigate the means to make TIG welding on these materials in atmospheric environment and analyse the effect of heat input on the properties of welds. It was found that it is possible to make TIG welds on these materials in atmospheric environment, provided that adequate protection is ensured on the face and root sides of welds. The increase of weld heat input coarsens the microstructure in melted and heat affected zones of both materials; increases also hardness in melted zones and tensile strength of welds, and there is not an obvious loss of ductility in heat affected zones.

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*Keywords:* Weld heat-input; tantalum; niobium; microstructure; tensile strength.

## 1. Introduction

Refractory metals, such as tantalum (Ta) and niobium (Nb), are increasingly applied in a variety of industries, such as chemical process industry, nuclear or even in medical device industry [1]. Niobium and tantalum have very high melting temperature and excellent corrosion resistance, but are very sensitive to oxidation at temperatures just above 300°C, which limits their use to moderate temperature applications or high temperature applications in non-oxidizing atmospheres. Welding of those materials presents several difficulties due to their great affinity to oxygen and nitrogen at elevated temperature [2]. Therefore, the welding of these materials should be conducted in vacuum or inert gas chambers in order to prevent the formation of brittle structures, using processes such as laser, electron beam or TIG, welding [3]. A significant

grain coarsening was reported in the heat affected zone (HAZ) as well as porosity in melted zone (MZ) of welds on commercially pure tantalum as the heat input increases in welds carried out with a CW Nd:YAG laser of 2.2 kW, using argon as shielding gas [4]. Significant grain coarsening in MZ and HAZ have already been observed in a previous study in TIG welds in tantalum and columbium (niobium) as well as porosity in MZ of Ta welds [5]. Pulsed Nd:YAG laser was used recently to weld pure niobium to Ti-6Al-4V with success but the study concentrates mainly on the effect of pulse current characteristics on penetration depth and tensile strength of the welds [6]. Electron Beam Welding (EBW) has been used for assembling single-cell and multi-cell cavities made of Nb; however, for small complicated parts of thin wall thickness, TIG welding can be advantageous [7]. However, when large equipment needs to be welded, it is not possible to weld in chamber; so, it is necessary to optimize the welding conditions outside the chamber. The purpose of this research is to optimize the manual TIG welding conditions in order to achieve

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similar microstructural and mechanical properties in welds on these materials performed into and out of the chamber. The results presented in this article refer only to welds performed in air using local shielding systems developed for the purpose in the company ARSOPI S.A., which is specialized in manufacturing equipment for chemical, petrochemical and nuclear industries.

## 2. Experimental Procedure

Similar welds were performed between plates of 250x80x2 mm size, in commercial Ta alloy R05200 – ASTM B 708-05 and Nb alloy R04210 – ASTM B 393-99, by manual gas-tungsten arc welding, using thoriated tungsten electrodes type EWTh2 and filler wire of similar chemical composition. The chemical composition and mechanical properties of the plates are shown in Tables 1 and 2. In fact they are made of tantalum and niobium commercially pure. Welds were done in air and shielded from the root and face sides with high-purity argon (99.996% Ar). Argon was supplied by specific diffusers developed for maintaining protection for the weld to cool to temperature below 200°C. Direct current connected for straight polarity, without any pulse, was used. Different heat inputs were used for each alloy, 1 and 1.7 kJ/mm for Nb and 1.3 and 2.5 kJ/mm for Ta, because of their different physical properties; for example, melting temperatures are very different (2468°C for Nb and 3017°C for Ta). The welds were radiographed to detect porosity or other defects and specimens for metallographic analysis, tensile and bend testing were removed transversely to welding direction. Metallographic specimens were polished according to conventional procedures but final polishing should be done using colloidal silica. Etching of Nb and Ta specimens was done in two similar steps: 25 mL lactic acid - 15 mL HNO<sub>3</sub> - 5 mL HF (48%) was used for 120s for the first step and 10 mL HNO<sub>3</sub> - 10 mL HF - 30 mL H<sub>2</sub>SO<sub>4</sub>, for 5 to 15s, for the second step. Vickers hardness tests were made at 0.5 kg for 15s on metallographic specimens. Tensile testing was done in specimens extracted transversely to the welding direction, with a cross section of 10x2 mm<sup>2</sup>, and the weld reinforcement was removed.

Tests were done in an Instron 4206 testing machine and an optical extensometer (ARAMIS) with digital image correlation (DIC) was used too.

Table 1. Chemical composition of niobium and tantalum plates.

Niobium R04210 (ppm)											
C	N	O	H	Zr	Ta	Fe	Si	Ni	Hf	Ti	Nb
39	34	83	3	<5	120	20	50	<5	<20	7	Rem.
Tantalum R05200 (ppm)											
C	O	N	H	Mo	Nb	Ni	Si	W	Ta		
7	32	18	2	<10	133	3	10	63	Rem.		

Table 2. Mechanical properties of niobium and tantalum plates.

Metal	Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)
Nb	225	127	54
Ta	245	161	59

## 3. Results and Discussion

### 3.1. Microstructure

All welds showed glossy appearance, revealing that protection was effective, but the morphology of the weld face is different between materials, as shown in Fig. 1.

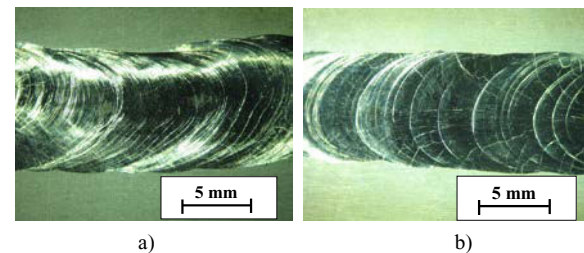


Fig. 1. Morphology of surface of welds on alloy: a) Nb (1 kJ/mm) and b) Ta (1.3 kJ/mm).

There was no internal porosity, or other defects in welds on both materials; however, there was substantial grain growth, both in MZ and HAZ of the welds, as illustrated in Fig. 2.

Although macrographs do not exhibit the same quality level because the polishing and etching of these materials is very difficult, it appears that the grain growth is more distinct in the MZ than in the HAZ. In the HAZ, the grain size decreases with increasing distance to the molten zone. Furthermore, it is apparent that the grain increases with increasing heat input (HI). The welds performed with higher HI exhibit some grains occupying the entire thickness of the weld (2000 μm), in contrast to welds performed with lower energy, which have in general at least two grains through plate thickness. Fig. 3 illustrates the

microstructure of HAZ and MZ of welds on Nb and Ta, done using the highest heat inputs. In HAZ of welds in Nb, Fig. 3 a), grain size (gs) is in the range 100 to 345  $\mu\text{m}$  while the average gs of base material is of 42  $\mu\text{m}$ . In MZ, Fig. 3 b), grains up to 1600x800  $\mu\text{m}$  were observed. For Ta, similar evolution was observed; in HAZ, Fig. 3 c), gs is in the range of 800  $\mu\text{m}$  to 1200  $\mu\text{m}$ , while base material gs is about 160  $\mu\text{m}$ . MZ, Fig. 3 d), displays gs up to 2000  $\mu\text{m}$ . Intragranular precipitates can be observed in MZ of all welds.

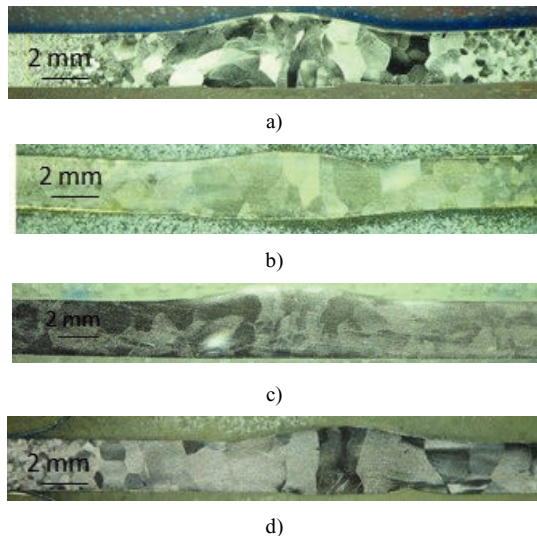
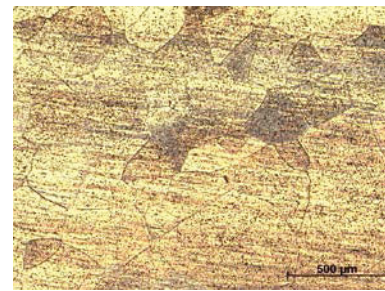


Fig. 2. Macrographs of welds performed on: a) Nb (1 kJ/mm), b) Nb (1.7 kJ/mm), c) Ta (1.3 kJ/mm) and d) Ta (2.5 kJ/mm).

### 3.2. Hardness

Fig. 4 illustrates the hardness fields in welds carried out in Nb and Ta plates, using different heat inputs. All welds exhibit a significant increase in hardness in the MZ, and a slight reduction in the HAZ. It is also observed that this increase in hardness is higher in welds performed using higher heat input. This increase in hardness can't be attributed to the variation of grain size since, according to the Hall and Petch law, the hardness and mechanical strength of metals decreases with increasing grain size [8]. This increase in hardness can be explained by the formation of precipitates in MZ due to the introduction of oxygen and/or nitrogen through the electric arc. Presumably, increasing weld HI, the oxygen and nitrogen contents, the volume fraction of precipitates and the hardness in MZs are increased. According to Kuwana [9] the oxygen and nitrogen absorption into melted iron is influenced by the welding conditions.



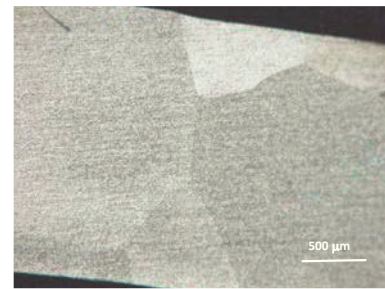
a)



b)



c)



d)

Fig. 3. Microstructure of welds on: HAZ of Nb (a), MZ of Nb (b), HAZ of Ta (c) and MZ of Ta (d).

Fig. 5 illustrates SEM/EDS analysis made in melted zone of a weld performed on Ta, using 2.5 kJ/mm. Precipitates of about 700  $\mu\text{m}$  size were detected in MZ, as shown in Fig 5 a). These precipitates are rich in oxygen as shown in Fig. 5 b).

Chauve *et al.* [10] mention that, in welds on zirconium, a reactive metal with characteristics similar to Nb and Ta, hardness is raised 10 points by 140 ppm increase in nitrogen content or by 200 ppm increase in

oxygen content. Fig. 4 also shows that there is some decrease in hardness in HAZ of both materials, certainly due to grain coarsening mentioned above. In these zones it was not detected any precipitates.

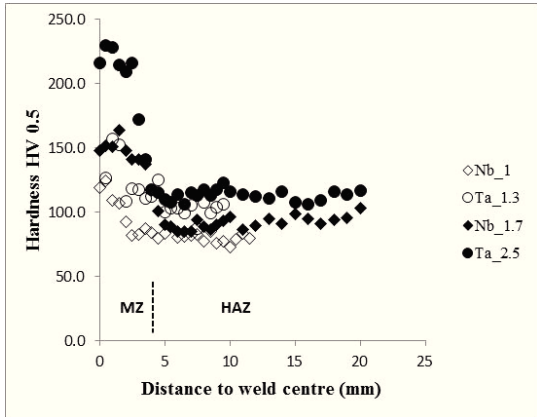


Fig. 4. Variation in hardness of welds done in Nb and Ta, using different heat inputs.

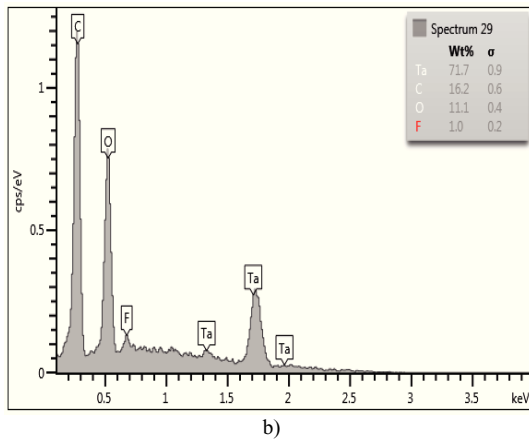
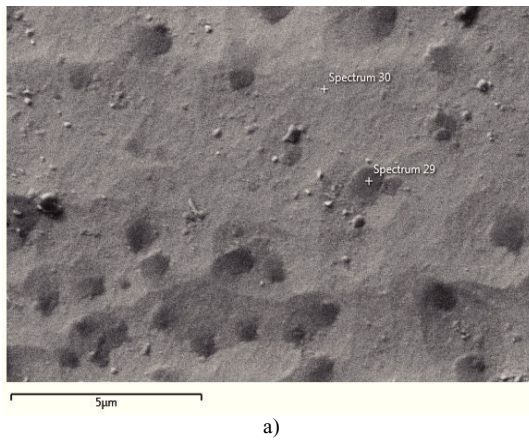


Fig. 5. Melted zone of a weld done on Ta with 2.5 kJ/mm: a) SEM image and b) EDS analysis of a precipitate.

### 3.3. Tensile strength

Table 3 illustrates the average tensile strength, elongation on fracture and efficiency of welds performed using different heat inputs, in Nb and Ta plates. The efficiency is defined as the ratio between the tensile strengths of the weld and its base material. It is obvious that, for both materials, the strength of welds increases with increasing heat input used in welding.

Table 3. Tensile strength of the welds.

Material	Heat input (kJ/mm)	Tensile strength (MPa)	Strain (%)	Efficiency (%)
Nb	1	136	6	60
	1.7	200	8.4	89
Ta	1.3	219	23	89
	2.5	237	13.4	98

The efficiency of the welds increases from 60% to 89% in the case of Nb, and from 89% to 97% for Ta. Table 3 also shows that the ductility of welds is much lower than its base materials, see Table 2. This is because plastic strain in tensile specimens concentrates in the HAZ, as shown in Fig. 6, the weakest region. In this case, the strain in HAZ is divided by the reference length of the test piece (50 mm), which leads to the low strain values listed in Table 3. Fig. 6 shows the local true strain fields in tensile specimens just after the maximum nominal stress. It is apparent the large plastic deformation suffered by the specimen before failure. Fig. 7 illustrates the fracture surface of two tensile test specimens taken from welds made on Nb and Ta. Both surfaces are only composed of dimples, attesting to the ductile character of fractures.

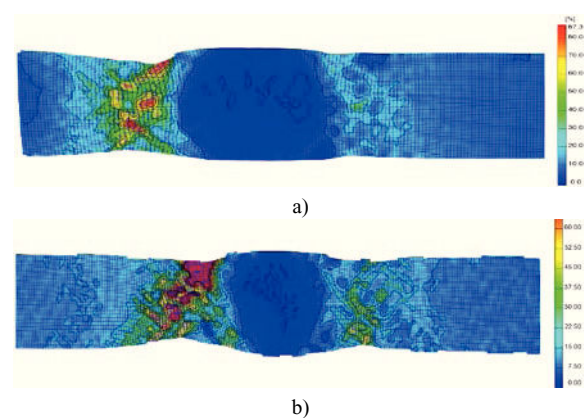


Fig. 6. Plastic deformation of tensile specimens removed from welds on: Nb (a) and Ta (b).

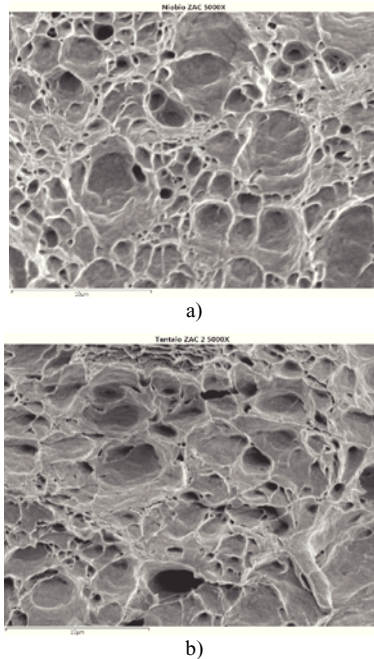


Fig. 7. Fracture surface in HAZ of welds on: Nb (a) and Ta (b).

#### 4. Conclusions

The work performed allowed the following conclusions:

- It is possible to perform TIG welding of niobium and tantalum in atmospheric environment, ensuring adequate protection with argon on the face and root sides of welds;
- The increase of weld heat input coarsens the grain size in melted and heat affected zones of welds in both materials;
- The increase of heat input increases hardness of melted zones and tensile strength of welds;

- There is no significant loss of ductility in the heat affected zone of welds.

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