Int. Journal of Refractory Metals and Hard Materials 50 (2015) 120–125

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Effect of vacuum arc remelting and processing parameters on structure and properties of high purity niobium

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ARTICLE INFO

Article history:
Received 12 August 2014
Received in revised form 25 November 2014
Accepted 6 December 2014
Available online 9 December 2014

Keywords:
Niobium
Vacuum arc remelting
Arc length
Thermomechanical processing

ABSTRACT

Niobium and niobium alloys are normally produced by vacuum arc remelting (VAR) process. In the present work, the effect of various process parameters such as arc voltage, arc current (melt rate) and fill ratio on arc stability during VAR process and solidification structure of remelted niobium ingot is investigated. The effect of thermomechanical processing of VAR ingot on microstructure and mechanical properties is also investigated. It has been established that arc voltage is a function of arc length. For melting of niobium in 110 and 150 mm diameter crucibles, the optimum arc voltage is in the range of 34–37 V. It has been observed that the arc voltage needed for melting niobium is much higher than that used for melting stainless steels, reactive metals and superalloys. It has also been observed that for a given arc voltage, fill ratio has no significant influence on the melt rate of consumable electrode. However, arc current has significant influence on the melt rate and consequently on the solidification structure of the ingot. Thermomechanical processing of the VAR ingot is shown to result in significant increase in room temperature strength and ductility. However, the processing temperatures do not have any significant influence on the tensile properties.

1. Introduction

Niobium exhibits a fascinating combination of attractive properties such as high melting point, low density, good fabricability, low ductile to brittle transition temperature and low thermal- neutron absorption cross section [1]. These properties make niobium a potential candidate material for applications in atomic power reactors, reentry vehicles, space power equipment etc. [2]. However, niobium suffers from poor resistance to high temperature oxidation, and active research is underway to overcome this limitation [3].

Historically, powder metallurgy methods involving high temperature vacuum sintering and carbon reduction have been extensively used to produce niobium metal [4]. However, these methods yielded niobium with higher oxygen contents which adversely affect its room temperature ductility, weldability and ductile-brittle transition temperature. Alumino-thermic reduction (ATR) and electron beam melting became the standard practice of producing high purity niobium since the early 1960s. Currently, industrial scale electron beam melting furnaces are available to produce niobium ingots of 300-500 mm diameter and over 2 m in length [5]. However, an electron beam melting process is mainly used only to refine impure niobium and has not been a very successful process for producing alloys based on niobium. This is because of the very high vacuum levels employed in the EB melting process which, while refining Nb may result in undue losses of alloying elements.

Vacuum arc remelting (VAR) process enables production of alloys based on reactive and refractory metals. Many of the commercial alloys based on niobium have been produced by vacuum arc remelting. The high temperature encountered in electric arc melting, the refining associated with protective vacuum cover, use of refractory free water cooled crucible and solidification in a water cooled mould enable production of high purity ingots with a desirable cast structure [6]. The success of the vacuum arc remelting process is essentially decided by the proper choice of the process parameters. The parameters which are crucial to maintain the melting process stable and to obtain good solidification structure are arc length, fill ratio and melt rate. However, the information available in literature regarding vacuum arc remelting of niobium and the effects of various process parameters on the quality of niobium ingot is inadequate.

In general, niobium is subjected to thermomechanical processing by hot forging followed by cold working. Hot forging temperature of commercial niobium is in the range of 650–950 °C. However, heating to such a high temperature leads to excessive oxidation of niobium. Further, oxygen, nitrogen and hydrogen can be picked up during heating and hot forging which make niobium brittle at room temperature. Therefore, niobium is protected against oxidation during heating, hot forging or intermediate annealing by employing protective coatings or inert gas atmosphere or by providing stainless steel jacketing. There is no information available in the literature on the influence of (i) different protection methods during heating and (ii) different thermomechanical

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http://dx.doi.org/10.1016/j.ijrmhm.2014.12.001

Table 1
Niobium ingots produced under different melt rates.

Ingot diameter (mm)	Average input power (kW)	Average melt rate (kg/min)
110	115	1.0
	165	1.5
	210	2.0
150	175	1.5
	220	2.0
	260	3.0

processing conditions on the structure and properties of VAR melted Nb ingot.

In the present paper, an attempt has been made to bring out the effect of various process parameters such as arc input power and voltage on melt stability and solidification structure of niobium ingots. The effect of different thermomechanical processing conditions on the structure and mechanical properties of VAR melted high purity niobium is also discussed in this paper.

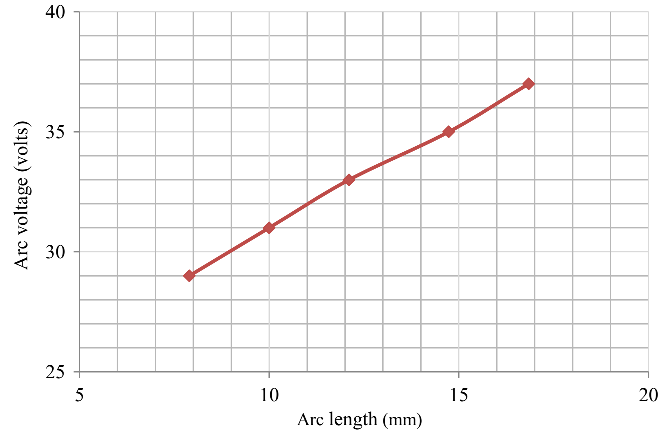

Fig. 2. Plot showing variation of arc length with arc voltage during VAR of Nb under vacuum.

2. Experimental procedure

A few VAR experiments were initially conducted in water cooled copper moulds of 60 to 150 mm diameter. Electron beam melted pure niobium rods were used as consumable electrodes. These experiments were conducted with different arc voltages with the following objectives:

(i) To establish the relation between arc length and arc voltage and
(ii) To optimize the arc length to produce and maintain stable arc.

Arc length is found to be an important process parameter during the VAR process. The distance between electrode bottom surface and top surface of the solidified ingot is taken as the arc length. Direct measurement of arc length is not possible during melting in the VAR process. Hence, this was measured during the VAR process by suddenly switching off the power and stopping the electrode feed. Then the electrode was moved downwards until it touched the ingot and the distance travelled by electrode was taken as arc gap.

Another set of experiments was carried out in 110 and 150 mm diameter water cooled copper moulds with different fill ratios (the ratio of cross-sectional area of consumable electrode to the cross-sectional area of the mould) and melt rates to study the effect on soundness and solidification structure of the ingots. Another experiment was conducted in 110 mm diameter mould wherein the power was switched off and the arc extinguished twice at different intervals, each time for a period of 4-5 min. The process was restarted using higher arc current initially to study the effect of power interruption on soundness of the ingot. The relevant process parameters (such as voltage, current, and vacuum levels used) were continuously recorded during all the VAR melts. Two trials were performed for each set of experiments.

Three pieces of 110 mm diameter and 470 mm length were cut transversely from the VAR ingot (110 mm diameter × 150 mm long) produced using the optimized process parameters. These pieces were subjected to different thermo-mechanical processing conditions as detailed in Table 1. The first ingot piece was coated with commercial grade (Deltaglaze FB 412) oxidation resistant coating and forged at 900 °C to 30 mm thickness. The second and the third ingot pieces were directly warm (250 °C) and cold (25 °C) forged to 30 mm thickness. Finally all the forged pieces were of 30 mm thickness. These forged pieces were stress relieved at 900 °C for 1 h and subsequently cold rolled to sheets of 2.0 mm thickness by using DEEMAG rolling mill. All the cold rolled samples were recrystallised at 1100 °C for 1 h.

The as-cast VAR ingots were tested for internal soundness using gamma-ray radiography (5 Curie Co-60 radioactive source) with an

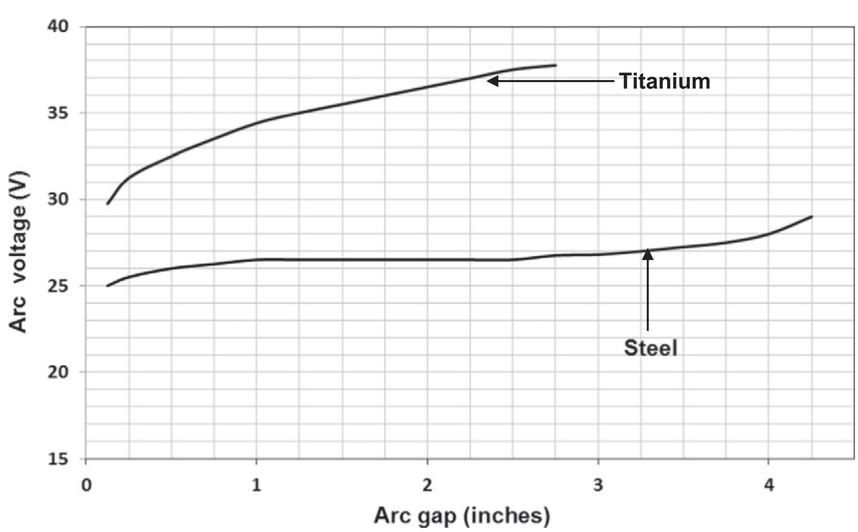

Fig. 1. Plot showing variation of arc length with arc voltage during VAR of Ti and steel under vacuum [7].