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OXIDE INCLUSIONS REMOVAL ON MICROSTRUCTURE PROPERTIES OF AS-CAST COBALT-BASED ALLOYS

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ABSTRACT

A cobalt-based metal alloying process was conducted with the addition of the non-metallic element boron (B) using Vacuum Arc Remelting (VAR). The process employed a water-cooled copper mold within an argon atmosphere. This research aimed to investigate the resulting microstructure and surface hardness values of the alloy. The metal melting rate during the VAR process was carefully controlled to achieve the desired microstructure and minimize defects, ensuring the production of high-quality alloys post-solidification. The process effectively facilitated the removal of oxide inclusions through flotation during remelting. The resulting alloy exhibited a dendritic microstructure characterized by large grain sizes. The average hardness value obtained for the alloy was 27.53 HRC.

Keywords: cobalt-based alloy; dendritic microstructure; solidification; Vacuum Arc Remelting (VAR).

ABSTRAK

Proses paduan logam berbasis kobalt dilakukan dengan penambahan elemen non-logam boron (B) menggunakan Vacuum Arc Remelting (VAR). Proses ini menggunakan cetakan tembaga berpendingin air dalam atmosfer argon. Penelitian ini bertujuan untuk menyelidiki struktur mikro dan nilai kekerasan permukaan yang dihasilkan dari paduan tersebut. Laju peleburan logam selama proses VAR dikontrol dengan cermat untuk mencapai struktur mikro yang diinginkan dan meminimalkan cacat, memastikan produksi paduan berkualitas tinggi setelah pemadatan. Proses ini secara efektif memfasilitasi penghilangan inklusi oksida melalui pengapungan selama peleburan ulang. Paduan yang dihasilkan menunjukkan struktur mikro dendritik yang ditandai dengan ukuran butiran yang besar. Nilai kekerasan rata-rata yang diperoleh untuk paduan tersebut adalah 27,53 HRC.

***Kata Kunci**: paduan berbasis kobalt; struktur mikro dendritik; solidifikasi; Vacuum Arc Remelting (VAR).*

1. Introduction

Metal casting processes are broadly classified into three types, including consumable molds, permanent molds, and special casting processes [1,2]. Consumable molds are divided into two types, including permanent patterns and expandable patterns [3]. Vacuum casting is a group of permanent patterns that really attracts attention [4]. Superior mechanical properties and high production rates are often realized in vacuum casting due to low mold temperatures. The casting process using the vacuum method that is commonly used is the vacuum arc remelting (VAR) process [5].

VAR process is one of the techniques for melting and casting of metal and alloys in a vacuum condition which then flows with argon (Ar) gas to produce metal ingots (anodes) [6-8] VAR was the first commercial melting process for super alloys. VAR process was used in the late 1950s to produce metals in the aircraft industry. Nickel (Ni)-based super alloys for aerospace applications are usually processed with VAR [9].

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Zirconium and niobium alloys used in the nuclear industry are also processed with VAR. Pure platinum, tantalum, and rhodium can be processed with VAR [10].

Casting, using the VAR method, is a manufacturing process that is commonly used to make metal-based biomaterials, such as cobalt (Co), titanium (Ti), and stainless steel (SS) metals [11-13]. Controlling the metal melting rate is very important in the VAR process to obtain the desired microstructure and minimize defects to produce superior quality products [14]. The VAR process generally uses a quenching cooling rate because the crucible is made of copper metal which is supported by water cooling media. Thus, the VAR process can be short and does not take time [15].

In recent decades, significant progress has been achieved in the development of biomaterials for medical applications, particularly in the synthesis of metal alloys designed for implants and orthopedic devices [16]. Cobalt-based alloys have attracted particular attention due to their unique combination of high mechanical strength, corrosion resistance, and good biological compatibility [17]. VAR method has become a valuable technique [18] in the manufacture of cobalt-based alloys, as it can increase the purity and homogeneity of the material while reducing material inclusions and defects. The addition of new elements is also continuously researched to obtain more favorable properties [19].

Based on this background, research was carried out on cobalt-based metal alloys with the addition of the non-metallic element, boron. Small amounts of boron addition (doping/ microalloying) can improve the non-cytotoxicity of cobalt-based alloys and boron is cheaper than cobalt. The method used is a melting process using VAR in Argon (Ar) atmospheric conditions. The VAR method in this research was used because it is considered cheaper than the powder metallurgy method [20]. The investigation discussed in this research is in the form of observing the microstructure and hardness values formed after removal of oxide inclusions.

2. Methods

The cycle that occurs during the VAR process lasts for one cycle, including the sample preparation process → sample input process into the VAR mold → vacuuming and back-filling the Ar atmosphere processes → melting process → solidification → flipped sample processes. Then, the remelting process is carried out again with the same stages of the initial cycle. Five repetition cycles in the VAR process was used to achieve composition better homogeneity [21,22]. VAR products generally are button ingots (coins) and rod ingots (bars), depending on the mold.

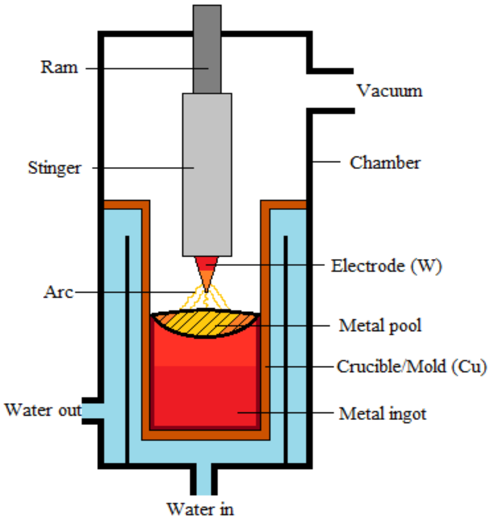

Figure 1. Schematic procedure of VAR

In this research, a Co-based alloy in ingot form was combined with pure Boron (B) elements (>99%), which is a semi-metallic element. Metals that can be processed or combined with a VAR machine are ingots and powder, or a mixture of ingots and metal powder. There are several steps that must be followed before using VAR method is carried out, such as calculating and determining the alloy composition, alloy preparation, and alloys weighing. Then, the sample is melted in the VAR chamber. After completing the melting process, the sample continues with the characterization process.

Metallographic observations of samples were carried out using an optical microscope. The as-cast samples were first sanded using a grinding and polishing machine under water, starting from coarse to fine sandpaper, that is 80, 120, 220, 320, 400, 500, 600, 800, 1000, 1200, 1500, 2000, 3000, 4000, and 5000 grid sandpaper. Then, continue with the polishing process using 1 µm Alumina micro polish. Next, the sample was soaked for 30 seconds in H_2SO_4 solution that mixed with methanol, with 1:10 volume ratio. Then, the etching process is carried out using an electrolyte etching test equipment (anode from SS, cathode from graphite/carbon) with a voltage of 6 Volts to accelerate corrosion. Then, the surface morphology of the sample was observed using an optical microscope.

Figure 2. Rockwell Hardness Testing

The sample's hardness was examined using a Rockwell hardness tester. The load given in this test is 150 kgf for 3 s with a diamond indenter, based on ASTM E18 standard. In this research, 10 hardness indentations were applied to obtain reliable data, consists of 5 tests at the bottom and 5 tests at the top. The results of the hardness values are recorded and averaged later.

3. Results and Discussion

The metal alloy melting process that has been carried out only takes 8 minutes in one cycle with the VAR process. The total time required to make a homogeneous ingot is 40 minutes because there is a 5× metal remelting process with the same cycle [23,24]. The final sample results are quite dense and homogeneous. The cooling rate after VAR took place quite quickly.

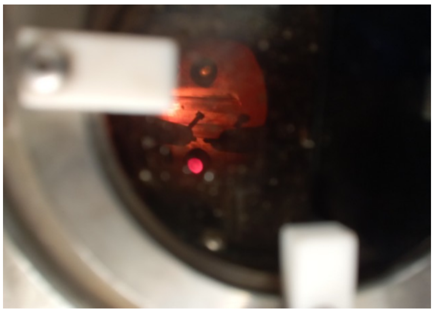

Figure 3. *As-cast* alloys after VAR process.

The main advantages of melting using consumable electrodes under vacuum conditions are: (i) removal of dissolved gases, such as hydrogen (H) and nitrogen (N); (ii) Minimizes trace content of undesirable elements that have high vapor pressure; (iii) improved cleanliness by removing oxides; and (iv) achieving ingot solidification directed from bottom to top to avoid macro segregation and minimize micro segregation [25].

Removal of oxide inclusions can be optimized during the VAR process. Oxide removal is achieved by chemical and physical processes. Less stable oxides or nitrides are thermally dissociated or reduced by other elements, such as carbon, present in the alloy and removed by conversion to the gas phase. However, non-metallic inclusions are very stable in super alloys. Removal of these inclusions occurs by flotation during remelting [26].

It was reported that oxide inclusions were formed and had separated from the main metal alloy. Oxides tend to float (density separation) because their density is lower than that of liquid metals. The vacuum used in the VAR process helps in reducing the amount of gas dissolved in the metal, which also helps encourage inclusions to rise to the surface. In addition, argon (inert gas) used in the VAR process also prevents oxide inclusions from dissolving with the metal melt [27].

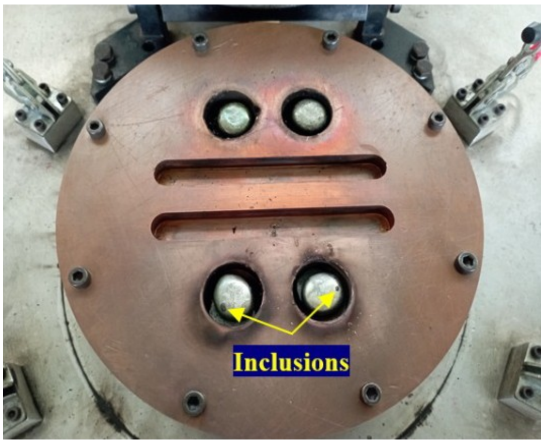

Figure 4. Metal oxide inclusions that float on top of the sample.

Oxide inclusions removal allows the avoidance of macrosegregation and reduces the degree of microsegregation in the final form of the alloy microstructure. Directional dendritic cellular solidification is essential for creating ingots that are both dense and homogeneous, and free from segregation. It is possible to achieve good mechanical properties. Simultaneously, oxide inclusions removal prevents internal flaws like porosity, and accumulations of nonmetallic inclusions [28].

Floating inclusions must be separated from the exterior surface of sample because it can degrade the properties of metal alloys [29]. Floating inclusions can be easily separated from the sample. One of the easiest ways is by scraping. The scraping process can be carried out using the tip of a lab spatula made by Stainless Steel (SS).

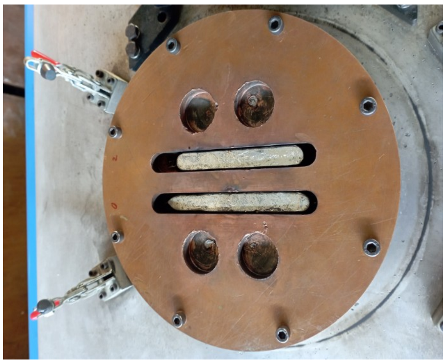

Figure 5. Co-based alloys with B addition rod ingot of VAR product.

Rod ingots can be formed from several button ingots that have been homogeneously produced, by melting them back in a mold. This is done to meet certain needs, such as to make tensile test samples. Rod ingot samples resulting from several button ingots produce more homogeneous shape when compared to making rod ingot samples directly from their initial shape (powder/ingot). This is because the button mold has a large enough space so that the blending and melting process can be more flexible [30].

Table 1. The hardness value (HRC) of Co-based alloys with B addition.

	Point 1	Point 2	Point 3	Point 4	Point 5
Top	26.2	26.0	27.3	26.8	24.0
Bottom	28.5	27.3	29.7	29.9	29.6
Mean			**27.53**		

Hardness value can be showed in Table 1. It can be showed that the bottom surface of the sample has a higher average hardness value than the top. This happened because the bottom of the sample is in direct contact with the water-cooled copper mold, so that the bottom sample will cool rapidly. Rapid cooling often produces residual stress in the metal due to differences in contraction rates between the inside and outside of the metal. This stress can provide an additional hardening effect [31].

The as-cast Co-based alloys with B addition microstructure is dendritic. The dendrites are shaped star-like. These dendrites are spread quite evenly on the surface of the sample. Additionally, there are also precipitates or sediment that appear. Precipitates arise as a result of the alloying process with other elements, because of B element (semi-metal) for this research, which occurs during the VAR process [32,33].

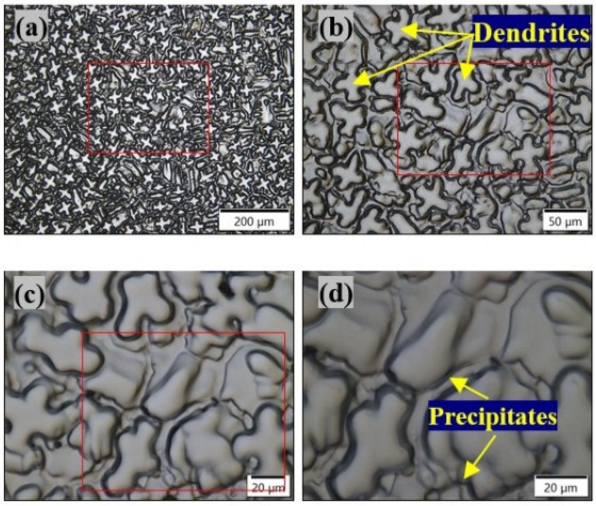

Figure 6. As-cast Co-based alloys with B addition microstructure with magnification (a) 20×; (b) 50×; (c) 100×; (d) 150×.

It can be seen that the microstructure of the sample is in primary dendrites form (stars-like), because columnar dendrites (elongated pine-tree-like) do not appear even at higher magnification. Dendrites appear when molten metal solidifies. Dendrites are a form of microstructure found in metal products resulting from the casting process. The shape and growth of dendrites affects the nature and properties of a metal alloys [34].

In the casting process, there are no significant restrictions on the direction of crystal growth as occurs in other more controlled metal processing techniques such as forging or rolling. This condition allows the dendrites to grow freely in any direction favored by local conditions. As the metal solidifies, the crystals have less time to form a uniform regular structure. As a result, the crystals grow rapidly in the direction of least thermal and kinetic resistance, resulting in a dendritic shape microstructure [35].

It was reported that the microstructure formed on the sample surface was oxide-free inclusions. Microstructure observations did not show porosity caused by oxide inclusions trapped in the metal surface. Oxide inclusions can have a negative impact on the mechanical properties and density of metals as it can become an initial point of failure when the metal is subjected to certain loads [36]. Oxide inclusions removal by floating on the sample surface is quite effective in clearing the sample surface from oxide exposure and failure.

The abrasive resistance of a metal depends on its surface hardness value, generally [37]. Hard metals have a denser and more tightly bound atomic structure, making it more difficult for other objects to penetrate. In the context of wear resistance, it means that a hard metal is better able to withstand surface deformation caused by friction or contact with foreign objects. In this way, wear that occurs due to contact or physical friction is minimized. This wear resistance property is needed for biomaterial applications so that it does not degrade quickly so that the metal has a long life [38,39].

Three main parts of the VAR: power source, cooling unit, and vacuum unit. In this research, the chamber is vacuum conditioned to a pressure of -100 kPa using a rotary pump vacuum unit. Then, a back fill process is carried out using Argon gas until the pressure returns to 0 kPa. This is done to avoid oxidation of the metal melt (Ar is an inert gas that does not react with the metal melt). Then, the melting process is carried out using remote mode (the current emitted by the electrode is controlled by a footrest, not constant), with a controlled current not exceeding 390 DC amperes for 2 minutes (cooling in the chamber is carried out after melting). The new ingot that has been obtained is turned over and remelted five times to ensure uniformity of the alloy in the sample [40]. More than one product can be cast in one process in VAR, depending on the crucibles in the mold.

The electricity source used to melt metal is a non-consumable electrode. Here, the electrode (Tungsten/W) acts as the cathode and the ingot acts as the anode. The VAR mechanism uses an electric arc that is struck between an electrode and alloys to produce emissions and sparks. This process is carried out in a chamber made of SS in a molded container made of water-cooled copper. Water circulation from the chiller (cooling unit) functions to cool the copper hearth and electrodes.

Figure 7. VAR machine with vacuum pump (left) and Ar gas cylinder (right).

The melting rate is fully controlled during the VAR process to obtain the desired micro-structure and minimize defects. The electrode gap should be small and constant. Medium and long electrode gaps (too far apart) can create non-uniform current distribution on the metal or mold surface and affect fluid flow in the mold. The diameter of the container is made larger than the electrode to prevent arcing between the electrode and the container wall. As a result, the electrode must be lowered as the melt consumes it.

Figure 8. Some metal elements in ingot form.

The main feature of VAR is continuous melting using consumable electrodes via direct arc (DC) under vacuum conditions. The molten metal solidifies in water-cooled copper molds. The basic design of a VAR furnace has remained largely unchanged over the years. However, significant progress has been made in the field of process control and regulation with the aim of achieving fully automated melting procedures. This in turn has a decisive positive influence on metallurgical properties of the products. Manufacturing homogeneous ingots with minimal segregation requires careful control of remelting parameters. Of these, the melt current density has the greatest influence on the melt bath geometry and solidification conditions [41].

4. Conclusion

Oxide inclusions removal in the microstructural properties of as-cast cobalt-based alloys has been successfully carried out using the vacuum arc remelting process (VAR). The vacuum process and argon gas used prevent oxide inclusions from dissolving in the metal. After the metal undergoes solidification, the oxide inclusions formed successfully float to the surface of the sample, which causes the final sample to be quite dense and homogeneous. These floating oxide inclusions are removed immediately as they can cause defects and material failure.

The final microstructure product in this research is primary dendritic (star-like shaped), due to oxide inclusions removal. This condition allows the dendrites to grow freely in all directions according to local conditions. The observed dendrite growth was evenly distributed in the microstructure observations. The shape and growth of dendrites can influence metal alloys properties.

Mechanical properties, in the form of hardness values, were achieved at an average of 27.53 HRC. Wear properties that occur due to contact or physical friction is minimized if the sample has high hardness. This wear resistance property is needed in biomaterial applications so that it does not degrade quickly and the metal has a long life. Further research that can be carried out on as-cast cobalt-based alloys for biomaterial applications is by testing performance in the body, such as toxicity testing, biocompatibility, corrosion testing, etc.

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