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Fabrication and mechanical properties of powder metallurgy tantalum prepared by hot isostatic pressing

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ABSTRACT

The fabrication process of a powder metallurgy (P/M) tantalum product with full density and fine microstructure was developed by using cold and hot isostatic pressing techniques. In order to increase the compact density and make the uniform density distribution, cold isostatic pressing (CIPing) of tantalum powders was conducted. Prior to hot isostatic pressing (HIPing), the CIPed billet was encapsulated and degassed to remove the contaminants in the container. After degassing, HIPing was performed twice and full densification of the tantalum powders was accomplished, regardless of powder size. The effect of processing conditions on the microstructure and mechanical properties of P/M tantalum billets was investigated. As the number of processing steps and temperature increased, the grain size of HIPed tantalum billets increased. Moreover, contrary to the Hall–Petch relation, the mechanical strength was increased in spite of increasing the grain size. This is because the oxygen content of the billets increased with rising in temperature and the number of processing steps. Therefore, in case of tantalum, it is found that the mechanical properties of tantalum may be highly influenced by the amount of interstitial elements, especially oxygen, rather than microstructural properties.

1. Introduction

Tantalum has been used in high strain rate applications such as shaped charges in warheads because of its high density (16.65 g/cm^3) and good dynamic ductility [1]. However, there are two constraints on applying in shaped charge armaments: the high-prices of raw materials and inconsistent microstructural variation in mill products [2]. Such restrictions may limit the wide application of tantalum and cause anisotropic properties in the final product where they are used [3]. Therefore, numerous recent studies have focused on reducing fabrication costs and eliminating inhomogeneous microstructure [4–6]. Equal channel angular pressing (ECAP) was applied to remove inhomogeneous microstructure of tantalum mill products, such as texture banding, leading to controlled microstructure of tantalum liners [4]. However, the process demands great effort and cost despite eliminating the inhomogeneity successfully. Powder metallurgy (P/M) techniques, instead of thermo-mechanical processing, may enable near-net shape (NNS) processing of tantalum to final products [7]. This NNS process has the potential to reduce the manufacturing and labor costs to produce a part. Furthermore, P/M processing promotes randomly textured products, thereby minimizing unexpected variations in mechanical properties.

The sintering of tantalum powders with full densification requires high temperatures and long processing times due to its high melting temperature and low thermal conductivity [8]. Moreover, unlike tungsten and molybdenum, tantalum has high affinity for interstitial atoms (O, N, C, and H); therefore, it should be consolidated under high vacuum atmosphere in a furnace with heating elements made of a refractory metal not a graphite. However, it is difficult for such conditions to be satisfied simultaneously, because under high vacuum and temperature conditions, metal heaters in a furnace would be damaged. Thus, several studies have focused on improving the sinterability of tantalum powders [8–14]. The addition of nickel enhanced the densification of tantalum powders, but the presence of the additional element may degrade the high strain rate properties [8]. Nearly full density, i.e., 95 wt% of the theoretical value, was achieved by spark plasma sintering at 1700 °C; however, the unwanted phases, like tantalum carbides, were formed by contamination from molds [9]. Nanocrystalline tantalum powders were used to enhance sinterability [10]; however, a high amount of oxygen due to their large surface area would lead to reduction in the dynamic ductility of tantalum. Hot isostatic pressing has been widely used to consolidate tantalum powders to prevent contamination and inhomogeneous microstructures. Lavernia et al. reported that the density of a HIPed tantalum sample was approximately 95.6% with an average grain size of 110 μm [11]. H.C. Starck developed a P/M tantalum billet by sintering, HIPing and additional plastic deformation [12]. Moreover, Bingert et al. and Boncoeur et al. also fabricated a

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HIPed billet with homogeneous microstructures and sound mechanical properties [13,14]. However, few studies have been investigated into the effect of HIPing conditions on microstructure and mechanical properties of tantalum.

In this study, the influence of the HIP process on the sintering behavior and microstructure of tantalum powders was investigated. The mechanical properties at room temperature were evaluated, and the relationship among processing conditions, microstructures, and interstitial elements was studied.

2. Experimental

2.1. Characterization of raw materials

The raw powders used in this study were obtained from two different sources: H.C. Starck (designated as TaA) and Ningxia Orient Tantalum Industry Co. Ltd. (referred to as TaB). The scanning electron micrographs and characteristics of the powders are shown in Fig. 1 and Table 1, respectively. The particle size distributions were measured by a laser diffraction method (Beckman Coulter LS 230 model). The particle morphologies of both powders were irregular, and the average particle size of TaA was larger than that of TaB. The chemical compositions supplied by manufacturers are shown in Table 2. The oxygen content of TaA and TaB powders, one of the important factors in determining the properties, was 257 and 550 ppm, respectively. The difference reflects the larger specific surface area of TaB compared with TaA.

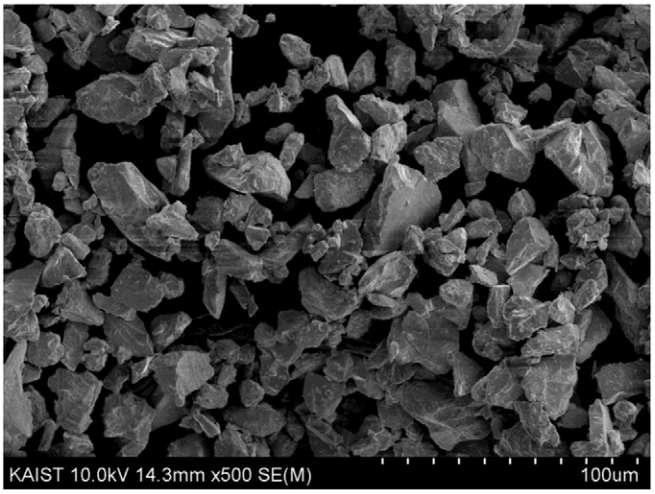

Fig. 1. Morphologies of (a) TaA and (b) TaB powders.

Table 1
Characteristics of raw tantalum powders.

		TaA	TaB
Average particle size (μm)		25.57	16.89
Particle size distribution (μm)	D_{10}	12.16	3.29
	D_{25}	17.53	5.51
	D_{50}	24.45	11.41
	D_{75}	34.63	21.65
	D_{90}	44.92	38.94
B.E.T. surface area (m^2/g)		0.0486	0.1501
Tap density (g/cm^3)		7.767	5.231
Manufacturer		H.C. Starck (GER)	Ningxia Orient Tantalum Industries Co. Ltd (CHN)

2.2. Consolidation

The powders were initially cold isostatically pressed (CIPed) at 2000 bar. The compacts were machined to cylinders; the green densities of the TaA and TaB powder compacts were 12.82 and 13.27 g/cm^3, respectively. They were placed into a titanium (Grade 2) container of 25 mm internal diameter, 100 mm length, and 1 mm wall thickness. Following compact loading, degassing was performed at 250 °C to remove vapor and contaminants in the powder. When the vacuum level in the capsule reached 10^{-2} torr, one end of the stem was crimped and welded. The encapsulated specimen was heated to 1500 °C, pressurized to 1000 bar, and maintained for 2 h under Ar atmosphere. Detailed conditions of hot isostatic pressing are shown in Table 3. The pressure and temperature were increased linearly up to the target values, where the pressure increased due to an increase in the temperature of the gas under constant volume. After HIPing, the container was removed by machining. The de-canned component was HIPed again without encapsulation to reach full density, as the pre-sintered product is already coherent, and the very small percentage of residual porosity ensures that there is little surface-connected porosity.

2.3. Evaluation of mechanical properties

The sintered densities of consolidated specimens were evaluated using Archimedes' principle, and their microstructures were also observed with an optical microscope. The tensile properties, such as yield, tensile strengths, and ductility, were characterized by the ASTM E 8 method. The oxygen content of the sintered components was measured by an elemental analyzer LECO® 836 Series. The effects of powder

Table 2
Chemical analysis of raw tantalum powders.

Element	TaA	(Unit: ppm) TaB
C	19	16
H	86	33
N	39	88
O	257	550
Fe	4	32
Ni	<2	8
Si	<7	40
Nb	<5	<30
Ti	–	<1
Mo	<4	17
W	6	10
Ta	Bal.	Bal.