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Molybdenum-based refractory alloys are conventionally prepared by vacuum arc melting technique on tonnage scale on a commercial basis and by powder processing route on a limited scale. However, in melting route, in view of high melting temperature of Mo (2650°C) and its reactive nature, power intensive steps like high temperature and high vacuum are employed during arc melting to melt and synthesize the alloys. Moreover, for this kind of refractory alloy, melting process becomes quite challenging to overcome the problem of segregation of minor alloying elements in view of their insigni cant quantities and large variation in the melting temperature with respect to major element. Hence, multiple melting trials are carried out to make the homogenized alloy. In some arc melting processes, an intermediate powder processing route is adopted to mix the alloying components well at room temperature in order to form homogenized consumable electrode, however, during melt consolidation by consumable arc melting technique again segregation recurs. So, overall, by following conventional melting or by a combination of powder processing and melting, it is di cult to achieve homogeneous alloy composition. As compared to the above routes, mechanical alloying (MA) is more advantageous as the entire operation of alloying is carried out at room temperature without application of any high temperature furnace or high vacuum system. In this process, repeated welding and rewelding of the elemental powders in a high energy ball mill produces a homogeneous distribution of alloying components and avoids many problems associated with melting and solidi cation [3].

In the present investigation, solid state powder processing by mechanical alloying technique was adopted to prepare TZC alloy. In this process, continuous milling and mixing of powder particles for prolonged duration was carried out to make homogeneous alloy at room temperature. MA powder was then fabricated into disk shapes by pressing and sintered to achieve high rate of densi cation. e alloy composition was evaluated by X-ray uorescence (XRF) analysis and corresponding elemental distribution in the alloy was investigated by electron probe microanalysis (EPMA). e structural evolution during the formation of the alloy was studied by X-ray di raction analysis (XRD) and corresponding powder morphological changes was investigated by scanning electron microscopy (SEM). e Microhardness pro le across the sintered samples was evaluated and optical as well as SEM characterization of the micro-structural features of the sintered alloy was studied. Transmission electron microscopy (TEM) was used to evaluate the carbide morphology in the resultant alloy.

Experimental

Materials

e main ingredient for the preparation of TZC alloy is Mo powder as its content is more than 98 wt% and other components are of minor elements of Ti, Zr and C whose combined content is not more than 2 wt%. However, Mo powder has tendency to form oxides easily during storage. Hence, in the present investigation, Mo powder used for TZC alloy preparation was freshly prepared in the laboratory by H_2 reduction of the oxide intermediates of Mo. e as-reduced actiaorl diswder us.162

Citation: Chakraborty and Krishnamurthy (2013) The Preparation of a Molybdenum Based High Temperature Refractory Alloy by Powder Processing Route. J Powder Metall Min 2: 113. doi:10.4172/2168-9806.1000113

Mechanical alloying

As-reduced active Mo powder obtained by hydrogen reduction was blended thoroughly with other elemental powders of Ti and Zr in the desired proportion using a Turbo-mixture. e blended powder was subsequently milled in a high energy 4 bowl planetary ball mill as shown in Figure 2 on 0.5-1 kg scale. Hard tungsten carbide balls of dia. 8 mm were used with charge to ball weight ratio as 10:1. Liquid medium of Toluene was used during milling to avoid any atmospheric contamination of Mo, Ti and Zr as these components are highly prone to oxidation. Toluene also served the purpose of carbon addition during the formation of TZC alloy. In order to evaluate the process of alloying, milling was interrupted periodically and a small amount of powder was collected for XRD analysis at every 5 hrs intervals. e vials were rotated at an angular speed of 300 rpm. e detailed experimental parameteŕo1(hr)6(s)112(f 30r)13(e r)11(w m)4(et)6(er) 745.5i ed (v)-3 howde5(l)-5()-8(va)-5(, T)16(n0.016 Tw T* [(w)-5(d ex65(l a)9(m

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Figure 16: SEM image showing carbide morphology in a typical arc melted TZC alloy.

and of polyhedral shape. e structural evolution of the milled powder by XRD analysis con rmed the formation of TZC alloy phase via crystallite re nement. TEM study further revealed uniform distribution of nano particles of carbides of Ti and Zr having size range of 3-5 nm in the matrix of TZC alloy. Faster sintering kinetics for MA powder was observed between 1500 and 1700 C.

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