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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, 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 insignificant 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 difficult 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 solidification [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 densification. The alloy composition was evaluated by X-ray fluorescence (XRF) analysis and corresponding elemental distribution in the alloy was investigated by electron probe microanalysis (EPMA). The structural evolution during the formation of the alloy was studied by X-ray diffraction

Experimental

Materials

The 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 reduction of the oxide intermediates of Mo. The as-reduced alloy powder was

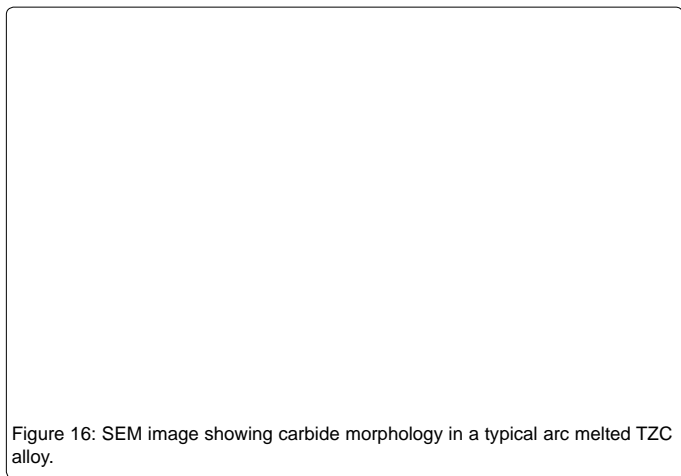
Mechanical alloying

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. The 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. The vials were rotated at an angular speed of 300 rpm. The detailed experimental parameters are given in Table 1.

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and of polyhedral shape. The structural evolution of the milled powder by XRD analysis confirmed the formation of TZC alloy phase via crystallite refinement. 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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