4th NANO TODAY CONFERENCE 2015

Comparative study of nano-TiO_2/Y_2O_3 dispersed zirconium based alloys by mechanical alloying and conventional sintering

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Abstract

Zirconium based alloys find wide variety of applications in high temperature, nuclear and chemical industries due to its good mechanical properties, excellent corrosion resistance and very low thermal neutron cross section. These alloys have high thermal conductivity and less thermal expansion coefficient than stainless steel which is necessary for elevated temperatures dimensional stability. Moreover, elevated temperature yield strength, creep and corrosion resistance of zirconium alloys can be increased by dispersion of nano-oxides. Compared to high temperature conventional melting method, preparation of zirconium based alloys by mechanical alloying followed by low temperature fast consolidation process is better in producing ultra-fine final structure and in improving strength properties.
The present study deals with the synthesis and mechanical property investigation of 1.0-2.0 wt.% nano-TiO_2/Y_2O_3 dispersed Zr based alloy with nominal composition of 45.0Zr-30.0Fe-20.0Ni-5.0Mo (alloy A), 44.0Zr-30.0Fe-20.0Ni-5.0Mo-1.0(TiO_2/Y_2O_3) (alloy B) and 44.0Zr-30.0Fe-20.0Ni-4.0Mo-2.0(TiO_2/Y_2O_3) (alloy C), synthesized by mechanical alloying (MA) followed by conventional sintering. In order to examine the microstructure, morphology and blending homogeneity the alloyed powders were subjected to X-ray diffraction (XRD), microscopic characterization by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Recrystallization behaviour of the final milled powder was studied by DSC. The average grain size of the as milled powder after 10 h of milling was 10-15 nm. The calculated activation energy of the base alloy A was 310 K J/mol. Consolidation of the milled powder was done by 0.998GPa uniaxial pressing and conventional sintering at 1673K (1400^{o}C). Finally, comparative study of the Zr-alloys (dispersed with TiO_2/Y_2O_3) was made in terms of hardness and wear properties. With increasing dispersoid content the hardness and wear properties increase. The maximum hardness observed for alloy C (with Y_2O_3dispersion) was 6.2GPA whereas with TiO2 dispersion it was 7.0GPa. In line of this, the alloys with TiO_2 dispersion showed better wear properties than Y_2O_3 dispersion.

Keywords: Zirconimum; Sispersuion; TEM; Mechanical properties

Zirconium alloys are the primary structural materials for nuclear and chemical sectors. Since 1960 Zirconium alloys are the principle cladding materials with excellent corrosion resistance, very low thermal neutron cross section and good mechanical properties [1--4]. Zirconium alloys have superior thermal properties compared to other traditional materials. The thermal conductivity of Zirconium alloys is 30% higher than stainless steel group of alloys and the linear coefficient of thermal expansion is one third the value of stainless steel which provide zirconium alloys superior dimensional stability at elevated temperature [5--7]. Moreover, elevated temperature yield strength, creep and corrosion resistance of zirconium alloys can be increased by dispersion of nano-oxides. Compared to high temperature conventional melting method, preparation of zirconium based alloys by mechanical alloying followed by low temperature fast consolidation process is better in producing ultra-fine final structure and in improving strength properties.

Present study deals with the synthesis and mechanical property investigation of 1.0-2.0 wt.% nano-TiO_2/Y_2O_3 dispersed Zr based alloy with nominal composition of 45.0Zr-30.0Fe-20.0Ni-5.0Mo (alloy A), 44.0Zr-30.0Fe-20.0Ni-5.0Mo-1.0(TiO_2/Y_2O_3) (alloy B) and 44.0Zr-30.0Fe-20.0Ni-4.0Mo-2.0(TiO_2/Y_2O_3) (alloy C), synthesized by mechanical alloying (MA) followed by conventional sintering. In order to examine the microstructure, morphology and blending homogeneity the alloyed powders were subjected to X-ray diffraction (XRD), microscopic characterization by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Recrystallization behaviour of the final milled powder was studied by DSC. Consolidation of the milled powder was done by 0.998GPa uniaxial pressing and conventional sintering at 1673K (1400^{o}C). Finally, comparative study of the Zr-alloys (dispersed with TiO_2/Y_2O_3) was made in terms of hardness and wear properties.

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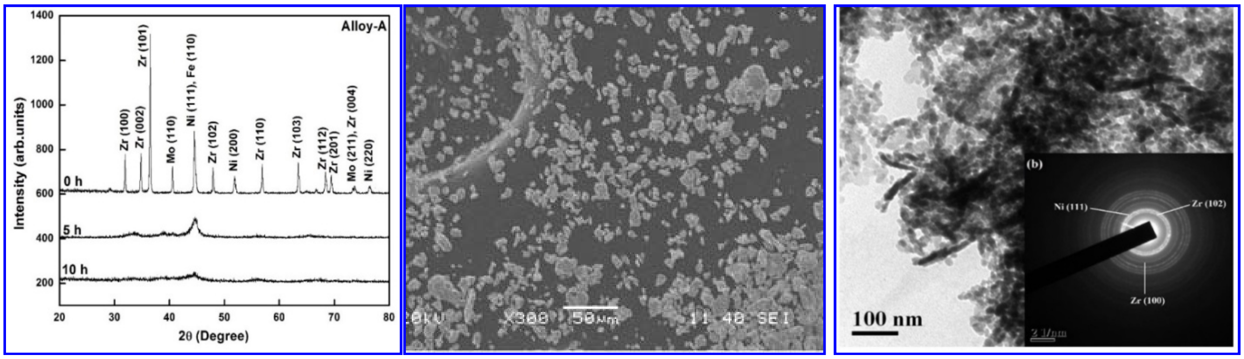
XRD analysis of the milled powders of different compositions obtained from different stages (0,5,10 h) of milling from both the mills shows that with increasing milling time elemental powders (Fe, Ni and Mo) slowly goes into solid solution and at final stage the alloys show single-phase Zr rich extended solid solution. With increasing milling time it was also observed that refinement of crystallite size and induced strain make the XRD peaks broader.

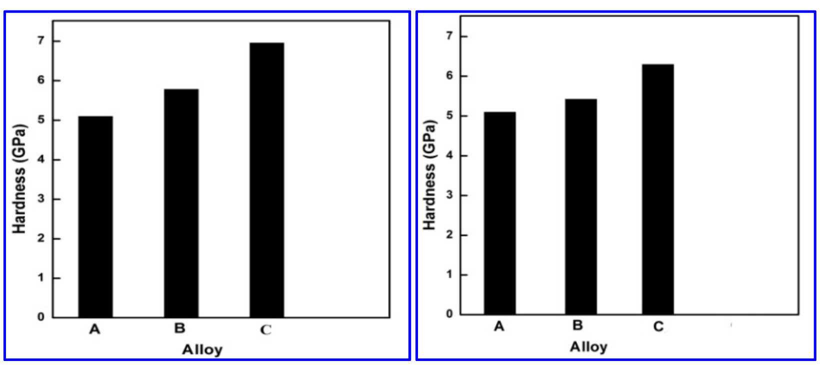


(a) (b) (c)

Fig. 1: (a) XRD patterns of milled, (b) powder morphologies of alloy A after 10 h of milling and (c) TEM image and corresponding SAD pattern of 10 h milled powder of alloy A

The SEM and Particle size analysis studies showed gradual refinement in particle size with increasing milling time. TEM analysis of final milled powders reveals 10-15 nm crystallite size.

Phase evolution of consolidated products by XRD analysis showed presence of intermetallic compounds like Mo_2Zr, $FeZr_2$, NiZr and $Ni_{11}Zr_9$ along with TiO_2/Y_2O_3 phase in Zr matrix. From bright and dark field TEM image and SAD analysis, presence of different intermetallics of 20-30 nm size along with TiO_2/Y_2O_3 (10-20 nm) throughout the matrix was confirmed. It was found that hardness increases with increase in dispersion of TiO_2/Y_2O_3 and maximum hardness of 7.0 GPa was recorded for TiO_2 dispersed C alloy. Wear test results display similar trend as that of hardness. The wear mechanism was found mainly abrasive in nature in all the samples.



(a) (b)

Fig. 1: Variation of hardness with (a) TiO_2 and (b) Y_2O_3 fraction.

From the present study it can be concluded that oxide dispersion can increase the hardness and wear properties of Zr based alloys and TiO_2 dispersion shows marginal better mechanical property over Y_2O_3.

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