Fabrication of nano ZrO\textsubscript{2} dispersed novel W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5} alloy by mechanical alloying and pressureless sintering

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Abstract: A high energy planetary ball-mill was employed to synthesize tungsten (W) based alloy with nominal composition of W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} (in wt. %) for 20 h with chrome steel as grinding media, toluene as process control agent (PCA) along with compaction at 500 MPa pressure for 5 mins and sintering at 1500°C for 2 h using Ar atmosphere. X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDS), elemental mapping and Transmission electron microscopy (TEM) was used to study the phase formation, microstructure of both milled powder and consolidated alloy. The crystallite size of W in W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} powder was 37 nm, 14.7 nm at 10 h and 20 h of milling respectively and lattice strain enhances to 0.54% at 20 h of milling. The crystallite size reduction is more at 10 h of milling and the rate drop beyond 10 to 20 h of milling. The intense improvement in dislocation density was evident upto 10 h of milling and the rate decreases between 10 to 20 h of milling. Increase in the lattice parameter of tungsten in W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} alloy upto 0.09% was observed at 10 h of milling owing to severe stress assisted deformation followed by contraction upto 0.07% at 20 h of milling due to formation of solid solution. The large spherical particles at 0 h of milling transformed to elongated shape at 10 h of milling and finer morphology at 20 h of milling. The average particle size reduced from 100 µm to 4.5 µm with the progress of milling from 0 to 20 h. Formation of fine polycrystallites of W was revealed by bright field TEM analysis and the observed crystallite size from TEM study was well supported by the evaluated crystallite size from XRD. XRD pattern and SEM micrograph of sintered alloy revealed the formation of NbNi, Ni\textsubscript{3}Ti intermetallic phases. Densification of 91.5% was attained in the 20 h milled and sintered alloy. Mechanical behaviour of the sintered product was evaluated by hardness and wear study. W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} alloy showed increase in hardness with decreasing load. The wear rate increases with increasing load due to higher abrasion effect at higher load.

1. Introduction
Highest melting point (3410°C), density (19.3 g/ml), excellent mechanical strength at elevated temperature, tensile elastic modulus (411 GPa at 20°C) and hardness of 9.8 GPa of tungsten (W) is fruitful for electrical, electronic, nuclear and space vehicle application [1]. Reduced workability and high ductile-brittle transition temperature (DBTT) (200-500°C) of tungsten imposes serious concern in its applications. Fabrication of tungsten based alloys in recent times is attributed to widen the application window and therefore to enhance the properties of W such as lowering of DBTT.

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Nanocrystalline tungsten alloys are potential candidates in this regard owing to enhanced mechanical properties [2, 3]. Mechanical alloying is a superior low temperature synthesis method of nanostructured materials by crystallite size refinement to improve the mechanical properties as compared to melt spinning and similar rapid quenching methods [4]. High-energy ball milling is a convenient alloying method due to easiness, mass production, improved productivity, comparatively economical equipment, and ability to fabricate a wide range of materials [5]. Ni addition is beneficial in improving the plastic flow property by alteration of dislocation core symmetry and also accelerates liquid phase sintering in case sintering temperature higher as compared to the melting point of Nickel (1455°C) [1]. Nb enhances the high temperature response of W [1] and Ti eases the densification of W, restricts the grain growth, however it increases the DBTT of W [6]. Dispersion of ultrafine particles at the grain boundary of W inhibits the grain boundary migration and subsequently improves the strength by dispersion strengthening mechanism [7].

The objective of the present research work is to reduce the sintering temperature of W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy by nanostructuring via mechanical alloy before sintering. Milled powder and sintered W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy was characterized by various characterization method.

2. Materials and methods
Elemental W, Ni, Nb, Ti (Sigma Aldrich, 99.5% purity, size: 50-100 µm), ZrO$_2$ (Sigma Aldrich, 99.8% purity, size: <100 nm) powders were milled in a planetary ball mill (Fritsch Pulverisette P5) with 300 r.p.m mill speed, chrome steel as grinding media and ball to powder weight (BPR) ratio of 10:1. Milled powders of 1, 5, 10, 15, 20 h were collected from the vial for characterization. The alloy composition and milling parameters are provided in table 1.

| Composition (wt.%) | Grinding Medium | Ball to powder weight ratio | Mill Speed (rpm) | Milling Duration (h) | Milling Medium |
|-------------------|-----------------|-----------------------------|------------------|----------------------|----------------|
| W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ | Chrome Steel | 10:1 | 300 | 20 | Toluene |

X-ray diffraction pattern (XRD) of the mechanically alloyed powders of various milling times and the sintered alloy was investigated by high resolution X- ray diffractometer (Make: Rigaku Japan, Model: Ultima IV) by using Cu-K$_\alpha$ radiation ($\lambda=1.541874$ Å) with scan speed of 10°/min and step size of 0.05°. XRD analysis data and JCPDS data bank was matched to identify the appearance of phases during mechanical alloying [8]. Peak position and peak broadening from the XRD pattern was used to evaluate the crystallite size and lattice strain [9].

The dislocation density induced in the milled powder was measured by:

$$\rho_d = 2\sqrt{3} \left( \frac{\varepsilon^2}{D \times b} \right)^{1/2}$$

Where, b is the burgers vector of dislocations, b = (a√3)/2 for the bcc structure, a= cell parameter = lattice parameter, D = crystallite size, ε = lattice strain, [10].

Precise lattice parameter calculation method was used to measure the lattice parameter from XRD pattern after stripping of K$_{α2}$ of XRD pattern [9]. The mechanically alloyed powder morphology at various milling time and the microstructure of the sintered alloy was studied by scanning electron microscope (SEM) (Make: JEOL, Model: JSM-6084LV). Powders milled for 20 h was dissolved in
acetone in a test tube and intensely stirred by ultrasonic vibrator. Few drops of acetone containing powders were placed on a carbon grid prior to TEM study. Crystallite size of W in 20 h milled W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} powder was revealed by high resolution transmission electron microscope (HRTEM) (Make : JEOL, Japan, Model- JEM 2100) study. A uniaxial press was employed to compact the 20 h milled powders into pellets of 10 mm diameter by 500 MPa pressure and 5 mins dwell time for uniform pressure distribution throughout the sample cross section. The compacted pellets were subjected to sintering at 1500°C for 2 h with constant Argon injection (rate: 100 ml/min) to avoid any chance of oxide formation. Microhardness of the sintered product was recorded by a Microhardness tester (Make: Leco, Model: LM248AT) under load of 50 gf, 100 gf, 500 gf, 1000 gf with10 sec of dwell time. The wear depth of sintered alloy was measured by a ball on plate wear tester (Make: Ducom, Model: TR-208-M1) under 20 N and 30 N load with sliding speed and time of 25 r.p.m and 10 mins respectively.

3. Result and Discussion

3.1. X-ray Diffraction (XRD) study of milled powder

XRD patterns of milled W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} powder at various milling times (1, 5, 10, 15, 20 h) is shown in Figure 1. Increase in broadening (full-width half-maximum) and decrease in the intensity of W diffraction peaks with progress of milling as evident from XRD pattern refers to crystallite size reduction of W and lattice strain build up [11]. Nb (BCC) forms continuous solid solution with W (BCC) with increase in milling time whereas Ni (FCC) and Ti (Hexagonal) shows limited solubility owing to difference in crystal structure, electronegativity and atomic radius as revealed from the presence of Ni, Ti peaks at 20 h of milling. Broadening was evaluated by Scherrer formula by eliminating the contributions the strain and instrumental broadening error [8].

![Figure 1. XRD pattern of W\textsubscript{79}Ni\textsubscript{10}Ti\textsubscript{5}Nb\textsubscript{5}(ZrO\textsubscript{2})\textsubscript{1} alloy at various milling time.](image)

Williamson–Hall equation was used to measure the crystallite size and lattice strain [12] as:

\[
\beta \cos \theta = \frac{0.94 \lambda}{D} + 4\eta \sin \theta
\]  

where, \(\beta\) is the full width half maxima (FWHM) or broadening, \(D\) is the crystallite size and \(\eta\) is the lattice strain. The intercept and the slope from the plot of \(\beta \cos \theta\) and \(\sin \theta\) is evaluated. The crystallite size (D) is measured after incorporating the \(\lambda\) value (1.541874 Å) in the intercept, whereas the slope corresponds to lattice strain. The change in crystallite size and lattice strain with milling time is presented in Figure 2. The minimum crystallite size and maximum lattice strain is recorded 14.7 nm.
and 0.54% respectively at 20 h of milling. The crystallite size reduction rate is substantially higher at 10 h of milling and the rate slows down between 10 h to 20 h of milling. The crystallite size refinement and increase in lattice strain is higher as compared to recently investigated W$_{80}$Ni$_{10}$Nb$_{10}$ alloy due to the presence of Ti which incorporates brittleness into the system and higher surface energy associated with nano ZrO$_2$ particles [13]. Presence of Ti at W/W grain boundary contributes in crystallite size reduction by alteration of grain boundary cohesive energy [14].

The dislocation density present in W$_{79}$Ni$_{10}$Ti$_{5}$Nb$_{5}$ (ZrO$_2$)$_1$ alloy shows rapid enhancement at 10 h of milling, though the rate of increase is restrained beyond 10 h of milling (figure 3(a)). Figure 3(b) shows that the lattice parameter of W in W$_{79}$Ni$_{10}$Ti$_{5}$Nb$_{5}$ (ZrO$_2$)$_1$ alloy enlarges upto 0.09% at 10 h and reduces upto 0.07 % at 20 h of milling. Pabi et al. have reported similar trend of Nb lattice enlargement in mechanically alloyed pure Nb [15]. The present investigation also displays a similar trend. Nb and Ni form substitutional solid solution with W matrix. The atomic radius of W (0.193 nm) is larger than Ni (0.149 nm) and Ti (0.176 nm) and smaller than Nb (0.198 nm). Therefore the ensuing atomic radius displays a drop after 10 h of milling [16]. Similar contraction of W lattice in W-Ni-Mo alloys is reported in recent literature [17]. The stress required for dislocation generation (Peierls–Nabarro stress) rises with crystallite size reduction which results in decrease in the rate of crystallite size reduction and dislocation density enhancement. Grain boundary softening beyond 10 h of milling also corresponds to decrease in dislocation multiplication. Higher stress aided deformation is responsible for initial expansion of W lattice at 10 h of milling. Formation of substitutional solid solution majorly by Nb with W and Ni, Ti with W to a lower degree results in contraction of W lattice beyond 10 h of milling.

**Figure 2.** Crystallite size and lattice strain variation of W with milling time in W$_{79}$Ni$_{10}$Ti$_{5}$Nb$_{5}$ (ZrO$_2$)$_1$.

**Figure 3.** Variation of a) Dislocation density and b) Lattice parameter of W in W$_{79}$Ni$_{10}$Ti$_{5}$Nb$_{5}$ (ZrO$_2$)$_1$ with milling time.
3.2. Microstructure study of milled powder

The particle morphology and size variation of the milled powder with increase in milling time is displayed in Figure 4 (a-d). The size of milled powder particles varies from ~100 µm to ~4.5 µm with the progress in milling. The powder particles are constantly work hardened, flattened and fractured as the milling time increases. The larger, spherical particle shape at 0 h transforms to elongated and finer morphology at 10 h and 20 h of milling respectively. The interparticle distance between W and solute atoms reduces with increase in milling time which results in enhanced atomic diffusion for the formation of alloy. Particle agglomeration to some extend is also observed at 10 h of milling (figure 4c). The elongated particle morphology at 10 h of milling is related with reduced flowability owing to enormous interparticle friction which contributes in lower green density [17].

Figure 4. SEM images of powder morphology of W_{79}Ni_{10}Ti_{5}Nb_{5}(ZrO_{2})_{1} alloy at different milling times: (a) 0 h, (b) 5 h, (c) 10 h, and (d) 20 h.

Figure 5 (a) shows the bright field TEM image of W_{79}Ni_{10}Ti_{5}Nb_{5}(ZrO_{2})_{1} powder milled for 20 h. The size of the crystallites of W at 20 h of milling is in the range of 10 to 20 nm which is nearly identical with the measured crystallite size from XRD analysis (figure 2). Formation of fine polycrystallites is evident from figure 5 (b). Indexing of SAD pattern (figure 5 (b)) shows the presence of W (222), Ni (222), Nb (321), Ti (203), ZrO_{2} (400) planes. The measured interplaner spacing (d) values of BCC-W is 0.091 nm for (222), Ni : 0.09 nm for (222), Nb : 0.089 nm for (321), Ti: 0.099 nm for (203), ZrO_{2}: 0.0909 nm for (400) respectively. The interplaner spacing (d) values matches well with the standard values of BCC-W from JCPDS data files.
3.3. Phase evolution analysis of sintered alloy

The phase formation in sintered W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy is investigated from the XRD pattern (figure 6). Formation of NbNi and Ni$_3$Ti intermetallic is evident from the XRD study. The strengthening of the matrix is contributed by the hard NbNi and Ni$_3$Ti phases as well by the presence of ZrO$_2$ particles. Similar phenomenon of NbNi intermetallic formation is also reported in recent literature [18]. However the intermetallic induces brittleness in the alloy which is responsible for lowering the ductility of the W matrix. (211) diffraction peak of W in milled powder (diffraction angle : 87.6713°) is moved to higher diffraction angle (87.6738°) in sintered alloy due to W lattice distortion primarily by Nb and to lesser degree by Ni and Ti. The broadening of XRD pattern decreases in the sintered alloy as compared to milled powder owing to increase in particle size and reduction in lattice strain during sintering. Any other unwanted phase formation is not observed from the XRD pattern.

3.4. Microstructure study of sintered alloy

Figure 7 (a) shows the SEM micrograph of W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy sintered at 1500°C for 2 h. The micrograph displays the existence of Ni (gray phase) and W matrix (bright phase). The intermetallic phase formation (NbNi and Ni$_3$Ti : dark phase) is related melting point of Ni (1455°C).
which is lower than the sintering temperature (1500°C). However the melting point of Ti (1668°C) is higher as compared to the selected sintering temperature it is expected that size reduction of brittle Ti during mechanical alloying decreases the melting point below 1500°C and contributes in liquid phase formation along with Ni. Evolution of intermetallic decreases the weaker W/W area fraction (contiguity). The identified phases are confirmed from EDS analysis (figure 7(b)) and elemental mapping (figure 7(c-h)) and the phases are in good agreement with phases revealed from the XRD study.

Figure 7. (a) SEM micrograph (b) EDS pattern (c, d, e, f, g, h) elemental maps of W, Ni, Nb, Ti, Zr and O of 20 h milled W_{79}Ni_{10}Ti_{5}Nb_{5}(ZrO_{2})_{1} alloy sintered at 1500°C for 2 h.

3.5. Densification study

Degree of densification is evaluated from the percentage ratio of sintered alloy density to the theoretical density of the sintered alloy. The density of the sintered alloy is measured by Archimedes’ principle [19] as:

$$\rho_s = \frac{W_a}{(W_{sat} - W_{susp})} \times \rho_w \frac{gm}{cm^3}$$  (3)

Where $W_a$ is weight of the sintered sample in air. $W_{sat}$ is the weight of the sample with all the open porosity saturated with water, $W_{susp}$ is the weight suspended in water. $\rho_w$ is the density of water. The %densification is measured from the percentage ratio of sintered density to theoretical density. The calculated densification of the sintered alloy records 91.5% (porosity: 8.5%) by compacting at 500
MPa pressure with 5 mins of dwell time. Table 2 shows the measured weight of the alloy at different condition for evaluation of %densification. The liquid phase formation during sintering improves the densification by closing of the open pores owing to enhanced grain boundary diffusion and capillary action of the liquid phase.

| Alloy Composition | Weight in air (gm) | Soaked weight in water (gm) | Suspended weight (gm) | Sintered density (gm/cm³) | Theoretical density (gm/cm³) | % Densification |
|-------------------|--------------------|-----------------------------|-----------------------|--------------------------|-----------------------------|-----------------|
| W₇₀Ni₁₀Ti₅Nb₅(ZrO₂)₁ | 0.8246              | 0.8249                      | 0.760                 | 12.705                   | 13.88                       | 91.5            |

3.6. Hardness study

The microhardness has been measured by the following equation as [20]:

$$HV = 1.8544 \frac{P}{d^2}$$

P is the applied load and d is the diagonal length of the indentation.

Change in hardness across the cross section of the W₇₀Ni₁₀Ti₅Nb₅(ZrO₂)₁ alloy with different load is shown in figure 8. Increase in hardness with reduction in applied load is due to considerable elastic recovery at reduced load and measurement error of the diagonal length owing to smaller size of indentation at reduced loads [21]. The hardness is measured on the W matrix phase, however the deviation in hardness value at a definite load is attributed to compositional heterogeneity across the cross section. The mean hardness at all loads is lesser than reported hardness of nanostructured W, as W is substituted by less harder Nb, Ni and Ti addition [1]. The measured hardness is comparatively less than Y₂O₃ dispersed W based ODS alloy owing to enhanced high temperature stability, hardness and strength imposed by Y₂O₃ dispersion [22].

![Figure 8](image-url)
3.7. Wear study

Figure 9 shows the change in wear depth with sliding distance traversed by the indenter on the sample surface. The sliding distance can be calculated as [23]:

\[
\text{Sliding Distance (S.D) = } \left( \frac{R}{60} \right) \times t \times 2\pi r
\]

(5)

R is the number of rotation per minute traversed by the indenter on sample surface, t is time in sec (600 s), r is track radius (4 mm) measured from the center of the sample to the track. The wear depth increases with increasing load as revealed from Figure 9. The phenomenon may be owing to removing out the intermetallic and ZrO₂ particles from matrix and W/W interface to the wear track throughout the abrasion and results in increased wear. The variation in wear depth profile throughout the contour is attributed to compositional heterogeneity and variance in hardness (figure 8). The increase in width of the wear track with increase in load as evident from figure 10 can be attributed to the process of three body motion which contributes to increased wear owing to enhanced abrasion at higher load [24].

![Figure 9](image)

**Figure 9.** Variation of wear depth of W₇₉Ni₁₀Ti₅Nb₅(ZrO₂)₁ alloy milled for 20 h and sintered at 1500°C for 2h.

Wear rate is defined as volume of wear per unit load and sliding distance. Archard equation has been used to measure the wear rate at highest wear depth for both 20 N and 30 N load [25]. The measured wear rate at maximum wear depth has been recorded 4.10 \times 10^{-15} m³/Nm for 20 N load and 5.71 \times 10^{-15} m³/Nm for 30 N load respectively.

![Figure 10](image)

**Figure 10.** Micrograph of wear track of W₇₉Ni₁₀Ti₅Nb₅(ZrO₂)₁ alloy at a) 20 N b) 30 N load.
4. Conclusion
The conclusions derived from the synthesis, assessment of microstructure, physical, mechanical behavior of the investigated alloy are as follows:

- Mechanical alloying is a potential method for fabrication of W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy powder.
- Crystallite size of W in W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy reduces to 14.7 nm at 20 h of milling.
- Lattice parameter of W in W$_{79}$Ni$_{10}$Ti$_5$Nb$_5$(ZrO$_2$)$_1$ alloy increases up to 0.09% at 10 h and followed by contraction of 0.07% at 20 h of milling.
- Formation of nanocrystalline BCC W of 10(20 nm in size at 20 h of milling is confirmed from bright field TEM image and corresponding SAD pattern.
- Evolution of NbNi, Ni$_3$Ti intermetallic phase after sintering is attributed to the formation of Ni and Ti base liquid phase at present sintering temperature.
- Increase in microhardness with decrease in load is related with lower indentation size and measurement error of the smaller indentation diagonals at reduced loads.
- Increased abrasion at higher load results in enhanced wear rate.

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