#### Fabrication and characterization of nano–Y<sub>2</sub>O<sub>3</sub> dispersed mechanically alloyed W-Ni-Nb for high temperature applications

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Abstract. The present research work deals with fabrication of nano- $Y_2O_3$  dispersed tungsten (W) based alloys with nominal composition of W79Ni10Nb10(Y2O3)1 (alloy A),  $W_{78}Ni_{10}Nb_{10}(Y_2O_3)_2$  (alloy B) and  $W_{72}Ni_{10}Nb_{15}(Y_2O_3)_3$  (alloy C) (all composition in wt.%) by mechanical alloying in a planetary ball mill and compaction at 500 MPa pressure followed by sintering at 1500°C for 2 h in Ar atmosphere. The milled powders at different milling time and the consolidated products have been investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), high resolution transmission electron microscopy (HRTEM) and energy dispersive spectroscopy (EDS). Minimum crystallite size and maximum lattice strain of 20.3 nm and 0.42% respectively is achieved in alloy C. Maximum sinterability, hardness, compressive strength of 93.38%, 6 GPa, 2.5 GPa with appreciable wear resistance has been achieved in alloy C owing to the lower crystallite size, presence of higher oxide dispersion (Y<sub>2</sub>O<sub>3</sub>) and NbNi intermetallic. High temperature behavior of the sintered alloys is studied in a raising hearth furnace at a temperature range of 800-1000°C. The activation energy of oxidation decreases with increase in Y<sub>2</sub>O<sub>3</sub> content owing to higher energy of Y<sub>2</sub>O<sub>3</sub> dispersoids. Alloy A shows superior oxidation resistance at 800-1000°C as compared to rest of the alloys. The investigated alloys shows superior strength and elongation as compared to recently investigated W-Y<sub>2</sub>O<sub>3</sub> alloys [1, 2].

Keywords: W-Ni-Nb alloy, Mechanical alloying, Oxide dispersion strengthening, Hardness, Oxidation.

- References: [1] R. Liu, Z. M Xie, T. Hao, Y. Zhou, X.P. Wang, Q.F. Fang, C. S Liu. Fabricating high performance tungsten alloys through zirconium micro-alloying and nano-sized yttria dispersion strengthening, J. Nucl. Mater. 451 (2014) 35–39.
  - [2] Ho J. Ryu, Soon H. Hong. Fabrication and properties of mechanically alloyed oxide-dispersed tungsten heavy alloys, Mater. Sci. Eng. A 363 (2003) 179–184.

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# **BRIEF OUTLINE**

- Aims and Objective
- Introduction & Background
- Materials and Method
- Result & Discussions
- Conclusions
- References

# **Aims and Objective**

- To fabricate nano Y<sub>2</sub>O<sub>3</sub> dispersed W based alloys through pressureless sintering.
- Investigate the physical, mechanical and high temperature behavior of the fabricated alloys.



- Extreme temperature (~1000-1500°C)
- High strain rate (10<sup>6</sup> s<sup>-1</sup>) and
- Hydrostatic pressure (2–6 GPa)

A. Upadhyaya, Mater. Chem. Phys. 67 (2001) 101–110.

### >Any other application?

- 1. Radiation Shielding
- 2. Aviation counterweights
- 3. High rigidity tooling components



#### **Die & Punch**



## >Why Tungsten?

### Pros.

- High Melting point (3420<sup>o</sup>C).
- High hardness (9.8 GPa), MOE=407 GPa.
- Good thermal conductivity (1.74 W/cm K), low co-efficient of thermal expansion.
- Low-activating metal in radiation environment with low sputtering yield.
   (W.F. Smith, McGraw-Hill, 1993)

### **Cons..** with Tungsten

High ductile brittle transition temperature (200-500°C). (J. L. Johnson, Sintering of refractory metals, Woodhead Publishing, Cambridge (UK) 2010, pp. 357–380).

### >Why alloy addition ?

- To Improve fabricability and effective utilization of Tungsten.
- Ni imparts liquid phase sintering (if sintering temp is higher than m.p. of Ni) and improve plastic flow properties.
- To improve the high temperature strength and lower the DBTT of W by Nb addition.
- Y<sub>2</sub>O<sub>3</sub> addition to reduce the grain growth and improve strength and hardness and wear resistance.

>Why Nanostructuring ?

- To lower the sintering temperature.
   (R malewar *et.al*, *J. Mater. Res., 22 (2007*)
- To improve the mechanical properties.
   [H. Glieter, Acta. Mater., 48 (2000)]



#### Mechanical Alloying Mechanism of MA



	Composition (wt.%)				
Alloy	W	Ni	Nb	Y <sub>2</sub> O <sub>3</sub>	
Alloy A	79	10	10	1	
Alloy B	78	10	10	2	
Alloy C	72	10	15	3	

- **Elemental powders of** W, Ni, Nb (purity 99.5%, Sigma Aldrich) initial particle of 100-150 µm •  $Y_2O_3$  (<50 nm).
- **FRISTSCH** planetary ball mill, WC vials and 10 mm chrome steel balls
- > 300 rpm, 10:1 (BPR).
- Foluene (PCA)

### Sintering Cycle



Equipment and Method	Parameters measured
XRD (X-Pert High Score, Origin, Powdll)	<ol> <li>Crystallite size, Lattice strain, Lattice parameter, Dislocation density, phase evolution of milled powder</li> <li>phase evolution in sintered product.</li> </ol>
SEM/TEM	1. Morphology, compositional, indexing of milled and consolidated product.
Archimedes's principle	Density/porosity measurement, Sartorius Density Measuring Kit
Hardness	Hardness and Elastic Modulus evaluation (MTS)
Ball on plate wear tester	Wear study (load = 20 N, time = 10 mins, speed= 25 r.p.m)
Instron-SATEC KN600	Compressive strength study at room temperature
Raising Hearth Furnace	High temperature behavior.

#### Wear Measurement:



#### **Ball on Plate Wear Tester**



**B** cos  $\theta = (0.94\lambda)/d + C \sin\theta$  (1), d= crystallite size, C= lattice strain, B= full width at half maxima. [B. D. Cullity, *Elements of X-ray diffraction, 1978.*]



## SEM micrograph of powder morphology of alloy A at different milling time (a) 0 h, (b) 5 h, (c) 10 h, and (d) 20 h.



## SEM micrograph of powder morphology of alloy B at different milling time (a) 0 h, (b) 5 h, (c) 10 h, and (d) 20 h.







**XRD of Sintered of alloys A through C** 





SEM Sintered of alloys A through C







						Spec	ctrum 33
)							
	···-						
	2	4	6	8	10	12	14
Scal	e 3878 cts	Cursor: 13	697 (1 cts)				kel/

Element	Weight%	Atomic%
ОК	24.92	64.85
YL	75.08	35.15
Totals	100.00	

#### **TEM image of sintered** alloy C $(3\%Y_2O_3)$ 21

- Surface energy increases, GB area increased, diffusion distance decreases with reduction in crystallite size.
- Improved GB diffusion enhance sintering kinetics and superior densification.

#### Microhardness





The interdiffusion co-efficient for W-Nb system increases with decrease in W content. [R. F. Hehemann, S. Leber, Trans. Met. Soc. AIME, 236 (1966) 1040-1044].







S	strength (max) (GPa)		Coarser grains : Yielding
Alloy A	1.82	13.86	92W−5.6Ni−1.4Fe-1Y <sub>2</sub> O <sub>3</sub> $\rightarrow$ H. J. Ryu et
Alloy B	2.35	19.43	al. Mater. Sci. Eng. A 363 (2003) 179– 184 $\rightarrow$ CS : 800 MPa
Alloy C	2.54	19.55	Lesser brittle W content

 $\Delta \sigma = 2G \epsilon f$   $\Delta \sigma$  = increase in yield stress, G = shear modulus,  $\epsilon$  = measurement of strain field, f = volume fraction of dispersed particles.

### FRACTROGRAPHY





- Deformation mismatch between matrix and particle, large stress concentrations.
- Stress Concentration at GB→
   Localize strain→ cracking under deformation.





G. Liu et al. Nat. Mater. 12, 344–350 (2013) in Mo-La<sub>2</sub>O<sub>3</sub> Increased  $Y_2O_3$  content at GB- $\rightarrow$  Increased Coalescence of particle $\rightarrow$  enhanced intense stress/strain concentration.

**TEM** image of sintered alloy C







## Activation energy for Oxidation

Alloy	Oxidation Temp (T)°C	I/T (K)	Oxidation rate (K)	ln K	Activation Energy (KJ/mol)
Alloy A	800	0.000932	72.52	4.283862	
	900	0.0008525	1682.32	7.427929	222.80
	1000	0.0007855	3457.577	8.148323	
Alloy B	800	0.000932	963.3957	6.870464	
	900	0.0008525	3956.664	8.283157	216.57
	1000	0.0007855	45389.01	10.72303	
Alloy C	800	0.000932	1306.36	7.175	
	900	0.0008525	7859.865	8.969525	199
	1000	0.0007855	43739.54	10.68601	

 $(\Delta m/S)^2 = K_P t + C \dots (1)$  $K_P = K_P \exp(-Q/RT) \dots (2)$ 

 $\ln (K_p) = -Q(1/RT) + C \dots (3)$ 



Schematic of oxidation mechanism in W-Ni-Nb-Y<sub>2</sub>O<sub>3</sub>.

# Conclusions

- Minimum crystallite size of 20 nm, maximum lattice strain and dislocation density of 0.42%, 27×10<sup>16</sup>/m<sup>2</sup> has been achieved in 20 h milled W<sub>72</sub>Ni<sub>10</sub>Nb<sub>15</sub> (Y<sub>2</sub>O<sub>3</sub>)<sub>3</sub> alloy.
- The % densification enhances with Y<sub>2</sub>O<sub>3</sub> addition owing to substantial reduction in crystallite size which enhances the sintering kinetics.
- The higher hardness (6 GPa), strength (2.25 GPa) of W<sub>72</sub>Ni<sub>10</sub>Nb<sub>15</sub> (Y<sub>2</sub>O<sub>3</sub>)<sub>3</sub> alloy is attributed to finer crystallite size and superior sinterability, higher content of Y<sub>2</sub>O<sub>3</sub> dispersion.
- %Elongation increases with increase in Y<sub>2</sub>O<sub>3</sub>, however exhibit marginal increase beyond 2% Y<sub>2</sub>O<sub>3</sub> content due to increased stress/strain concentration resulted from agglomeration of finer Y<sub>2</sub>O<sub>3</sub> particles during sintering.

## Conclusions

- Increase in Y<sub>2</sub>O<sub>3</sub> content promotes intergranular fracture during compressive loading as coarser Y<sub>2</sub>O<sub>3</sub> particles enhances the stress/strain and facilitates interfacial debonding.
- Superior oxidation resistance is achieved in lower Nb containing alloys and upto 2% Y<sub>2</sub>O<sub>3</sub>.
- W<sub>72</sub>Ni<sub>10</sub>Nb<sub>15</sub> (Y<sub>2</sub>O<sub>3</sub>)<sub>3</sub> alloy shows poor oxidation resistance due to increased difference in molar volume and thermal expansion co-efficient of higher content of oxides which results in spallation and cracking of oxide layer.

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