International Journal of Scientific Research & Engineering Trends
Volume 6, Issue 1, Jan-Feb-2020, ISSN (Online): 2395-566X

Ultrasonic Technique for Physco-Mechanical Evaluation of Tungsten Alloys by Taguchi Technique

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Abstract **- Nine versions of the basic Tungsten heavy alloy "90W–7Ni–3Co "were investigated for optimal characteristics. These versions of the WHAs were manufacturing via powder metallurgy (P/M) technique. Involved PM preparation parameters together with the additions of specific percent of the Nano-particles of graphene were nondestructively evaluated. Study showed the effectiveness of using Taguchi Technique in representing and analyzing experimental results. Each alloy version has its own specific chracteristics, where influential primary affective parameter was ranked.**

Keywords- **Tungsten heavy alloys, NDE, Ultrasound technique, Taguchi technique, Grapheme, Powder Metallurgy.**

I. INTRODUCTION

Tungsten and their alloys are used widely across industry for their unique specific characteristics including high melting point, high conductivity, hardness, density and durability [1]. Tungsten heavy alloys (WHAs) are ideal for high-density applications [2, 3] or for use in radiation shielding. Heavy metal tungsten alloys are 90% to 97% pure tungsten in a matrix of nickel and copper, nickel and cobalt, or nickel and iron. The addition of these alloying elements improves both the ductility and machinability of these alloys over non-alloyed tungsten [3]. Here, the authors did tray to optimize the characteristics of nine versions of WHAs; which are based on the heavy Tungsten alloy "90% W-7%Ni-3%Co". NDE technique was used in such evaluations.

II. DESIGN OF EXPERIMENTS

Preparation of the proposed Tungsten alloy versions are involving THREE main parameters; such as the process control agent (PCA), the Sintering Temperature, and the percent weight of the added Nano-particles of graphene. Table I, lists these different PM preparation parameters together with their proposed levels.

Table I: PM involved parameters with their levels.

Parameters	Level		
	1	2	3
Process Control agent (PCA)	Wax	Stearic Acid	Mineral Oil
Sintering Temp. (˚C)	1450	1475	1500
Graphene, wt.%	0.0	0.25	0.50

For full understanding of the impact of each parameter at specific level, will be time consuming and costly. Applying Taguchi technique (TT), would solve this problem and minimize the required number of runs and to optimize the test outcome. The application of Taguchi technique will ensure the optimization of combined parameters and factors with robust experimental design. Table II, lists the proposed experimental design by applying TT, where only nine experimental runs are proposed using an orthogonal array of L9, for optimized and robust experimental design.

Table II: The proposed Taguchi Orthogonal L9- Design.

WHAs Version ID	Sintering Temperature °C	Process control agent	% Graphene
1	1450	Wax	0.0
2	1450	Stearic Acid	0.25
3	1450	Mineral Oil	0.50
4	1475	Wax	0.25
5	1475	Stearic Acid	0.5
6	1475	Mineral Oil	0.0
7	1500	Wax	0.50
8	1500	Stearic Acid	0.0
9	1500	Mineral Oil	0.25

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III. EXPERIMENTAL

The NINE optimized experimental runs; based on the basic WHAs of "90W- 7Ni- 3 Co", were prepared in accordance with the conditions listed in Table 2. These Taguchi proposed NINE experimental runs, are suggested by the authors to be new versions for the basic alloy of "90W- 7Ni- 3 Co", with different chemical compositions and were fabricated powder metallurgy (P/M) technique (milling, cold compaction and vacuum sintering); where complete deails are found elsewhere [4].

The powders were blended in a ball mill (3:1 ball to powder ratio), and reduced at 700oC in H2 atmosphere for 6 Hrs. The reduced powders were cold iso-statically compacted into short prismatic pellet of 15*12 mm base cross-section and 10 mm height. As illustrated in Fig 1. Sintering of the pellet was carried out in according to Table II in vacuum, for Hr, with a heating rate of 5oC/min, following the sintering cycle shown in Fig 2.

Fig.2 Sintering cycle for tungsten heavy alloy version no 7, sintered at 1500 oC.

These PM fabricated Tungsten Heavy alloy (WHAs) versions were evaluated for their characteristics in many aspects. These characterizations are including mainly Metallographic examination (i.e. SEM and EDX analyses for their microstructures and grain size), and Non-destructive evaluation (NDE) using ultrasonic technique (UT) for their Mechanical and Physical properties. Ultrasonic technique (UT) was used, as a non-destructive evaluation technique [5] to define both Physical and Mechanical properties. Such NDE using UT is based of measuring elastic wave propagation velocities; longitudinally (VL), and along the outer surface (VS) of each WHAs alloy version. Longitudinal and shear probes were used on these acoustic measurements – see Fig 3.

Fig.3. The ultrasonic flaw detector device (USN 60 model), with Longitudinal (L) and Shear (S) Probes.

These two measured ultrasonic propagation velocities; V_L and V_S are listed in Table III, and were used to determine nondestructively the following mechanical properties [6,8]:

- The Modulus of Elasticity: $E=\frac{\rho * V_s(3V_l^2-4V_s^2)}{V_l^2-V_s^2}$
- The Modulus of Rigidity: $G = \rho * V_s^2$
- The Bulk Modulus: $B = \rho(V_l^2 - \frac{4}{3}V_s^2)$
- The Poisson Ratio: $\upsilon = \frac{1-2(\frac{V_S}{V_L})^2}{2(1-(\frac{V_S}{V_L})^2)}$

Table III. Experimentally measured Ultrasonic wave propagation Velocities for different WHAs alloy versions (±2.4% Uncertainty).

WHAs Version No	Longitudinal velocity, V_L (m/s)	Shear velocity, V_S (m/s)
1	5226	2853
2	5221	2869
3	5322	2912
4	5403	2879
5	5298	2950
6	5242	2882
7	5110	2746
8	5283	2886
9	5326	2869

IV. RESULTS AND DISCUSSION

Nine Tungsten Heavy alloy versions based on the basic one "90W-7Ni-3Co" were optimized using Taguchi method. These WHAs were fabricated using PM technique, and non- destructively evaluated (NDE) using ultrasonic Technique (UT). The main purpose is to enhance the performance characteristics of such alloy by adding the Nano-particles of graphene together with varying the preparation PM parameters of Sintering Temperature and its process control agent.

Figure 4 illustrate the SEM micrographs of the proposed nine sintered samples or WHAs versions of “90W-7Ni-3Co” WHAs. The dark grey areas in these micrographs represent the binder material, while the white grey particles are the pure tungsten and the black colour represented the graphene as shown by arrow. It is noticed the microstructures are homogeneous.

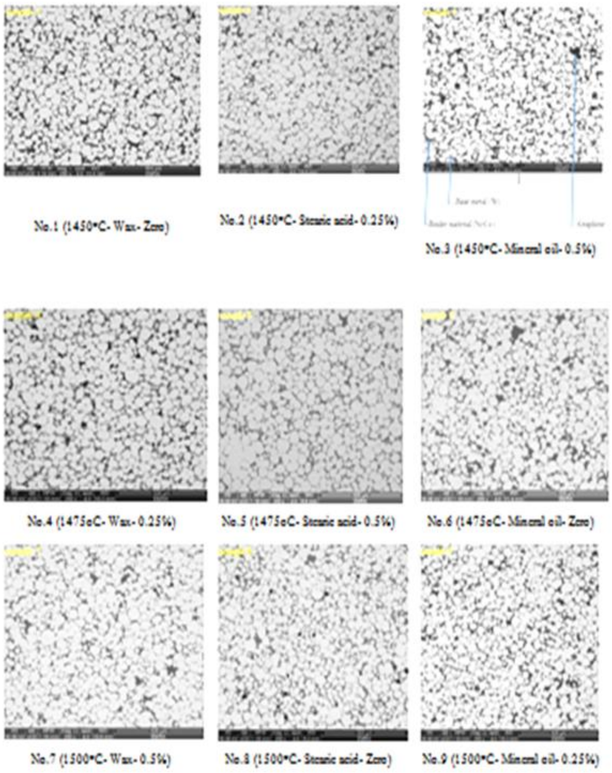

Fig.4. SEM micrographs for the WHAs “90W-7Ni-3Co” Nine versions.

As an example for the main constituents of the resulted fabricated WHAs versions, is shown in Fig 5, while their statistics are listed in Table4.

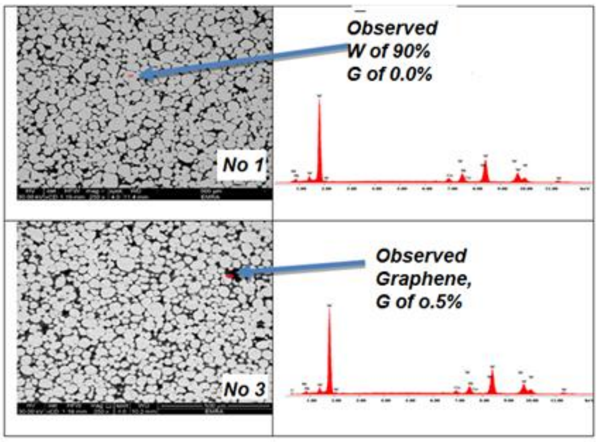

Fig.5. The EDX analysis for specimen versions No. 1 and No. 3.

Table IV. Elements contents of the proposed alloy versions No 1 and No 3 using.

Proposed alloys of “90W-7Ni-3Co”						
Alloy Elements	WHAs version No. 1			WHAs version No. 3		
	Actual	Observed	% error	Actual	Observed	% error
W %	90	87.72	2.53	90	87.60	2.66
Ni %	7	8.49	21.28	7	6.63	5.29
Co %	3	3.79	23.66	3	2.55	15
G %	Zero	Zero	zero	0.5	0.22	5.6
Total %	100	100		100	100	

Measured Vickers macro hardness, Grain sizes and density for the different tested WHA alloys versions are listed in Table V. Higher hardness value was observed for WHA No 5 (1475oC Sintering Temp, Stearic acid and 0.5%Wt graphene), while smaller grain size was noticed for WHA version No 8 (1500oC Sintering Temp, Stearic acid and 0.0%Wt graphene), finally fine density was resulte for WHA No 3 (1450oC Sintering, Minral oil and 0.5%Wt graphene). These conditions of PM preparation for WHA version can be a guide lines for a customized the Tungsten alloy "90W-7Ni-3Co".

Table V: Measured Vickers macro hardness, Grain size, and Density for different WHA alloy versions.

Alloy version /Specimen Number	Average Vickers Hardness	Average Grain Size	Actual Density (Kg/m^3)
1	339	42	15172.52696
2	379	40	14477.91077
3	301	44	13287.36842
4	352	48	14509.98497
5	379	42	13971.73913
6	360	45	14574.43609
7	317	37	13819.11381
8	323	36	15213.95076
9	289	38	13951.38662

As for the non-destructive evaluation for elastic modules, especially the modulus of elasticity, the Graph of Fig 6 represents both of the NDE values and the destructive (DT) measured values using foil-type strain gages. To some extent, there is a general agreement between destructive and the non-destructive values.

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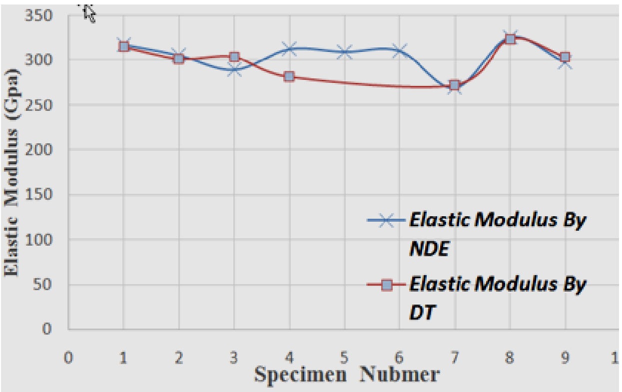

Fig.6. Variation of Elastic Modulus for different WHAs.

It is noted that, the maximum value reaches 326 GPa for specimen 8 (1500oC Sintering Temp, Stearic acid and 0.0%Wt graphene), and a minimum value of 265 GPa for WHA version No 7 (1500^{o}C Sintering, Wax and 0.50 %Wt graphene).

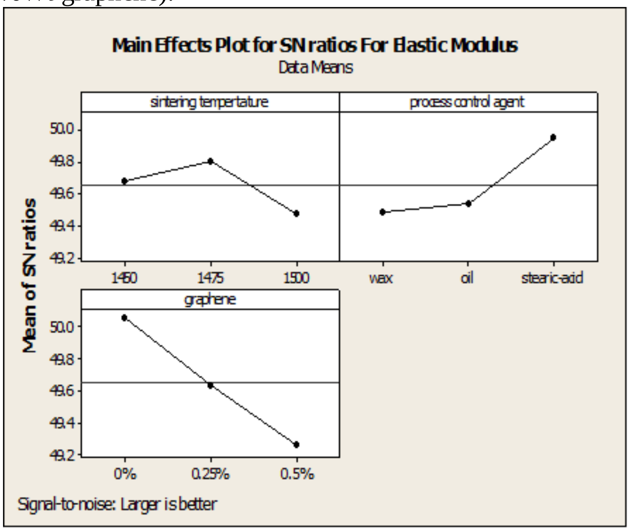

Fig.7. The Taguchi SN- index for modulus of elasticity the proposed alloys.

These NDE results are in good agreement with the Taguchi – SN indices; which gives a confidence status with the creditability of Taguchi technique as a good DOE tool.

V. CONCLUSIONS

The main conclusion of this work, can be summarized in Table VI, with emphasize on the ranking order of each contributed parameter on the resultant optimized yield. This is another good tool offered by the Taguchi technique as a DOE tool. The following points can be inferred:

1. The addition of Graphene has the primary order in enhancing all the Elastic modulus, the Bulk modulus and the Shearing modulus.
2. As for sintering temperature, it occupy the first influential factor, that enhancing some alloy characteristics such as making higher Hardness, lower Poisson Ratio and finer Grain Size.
3. As for the Relative Density, the PCA did show a stronger effect in producing a denser structure.

Table VI: Taguchi Defined Ranking order for the most Influential Parameters upon their characteristic outcomes.

Taguchi mean value (optimum)	Ranking order		
	Graphene %Wt	PCA	Sintering Temperature ($_0$C)
Elastic Modulus (higher)	1	2	3
Bulk Modulus (higher)	1	2	3
Shear Modulus (higher)	1	2	3
Vickers Hardness (higher)	3	2	1
Poisson Ratio (smaller)	3	2	1
Grain size (smaller)	2	3	1
Relative density (higher)	3	1	2

Another Taguchi technique results is the SN-index for the modulus of elasticity – see Fig 6. Since, a material alloy with higher or larges values is preferable for some applications, a condition of Sintering temperature at 1475^{o}C, with PCA of Stearic acide and zero percent weight of graphene should leads to this required characteristic (i.e. to some extend WHA No 5). Although WHA No 5, includes a 0.5% Wt grapheme not a zero ratio, but if one feed these condition back to Taguchi technique it will predict a value of As one can noticed, that different elastic moduli of Elasticity, Bulk and shear are mainly dependent to some extend on added the Nano-particles of Graphene For the graphene content it has a big influence effected on the elastic modulus, bulk modulus and shear modulus. While the sintering temperature has the same rank of big influence of the Vickers hardness, Poisson ratio and grain size.

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