DOI: 10.1515/amm-2017-0286

V. VISHNUH*, S. SUDHAKAR*, K. TAMILARASU*, P. PRABHAKARAN*[#], R. RAJASEKAR*

EXPERIMENTAL ANALYSIS OF NITROGEN AS AN ALLOYING ELEMENT IN WC9 GRADE STEEL

In the present work the effect of nitrogen on WC9 alloy at various weight percentages was analyzed and tested for their microstructural and mechanical properties. The nitrogen was added at 0.05, 0.10, 0.15, 0.20 and 0.25 wt. % in the solid form as nitrided ferrochrome to WC9 alloy. The samples were heat treated by solution annealing process at a temperature of 1100°C for 5 hours to improve the austenitic formation. Microstructures and mechanical properties such as tensile strength, yield strength, hardness, % elongation and % reduction of WC9-N alloy were examined. It was observed that increasing nitrogen wt. % increases the mechanical properties. The obtained mechanical properties were compared with base WC9 and C12A grade steel, where it was found to be replacement for C12A grade steel at its composition at lower end. The material cost analysis for WC9-N and C12A grade steel was done and both were compared.

Keywords: nitrogen alloying, WC9 steel, solution annealing, mechanical properties

1. Introduction

In this paper, we defined the effect of nitrogen in low carbon alloys in which the carbon content was about 0.05-0.25 wt. % of steel. Low carbon steel had excellent formability, weldability, machinability, hardenability but less ductile in nature. Therefore it was used in sheet manufacturing, piping, pressure vessel manufacturing etc. In low carbon steel, WC9 grade was incorporated to the addition of nitrogen in varying compositions. The solubility of nitrogen mainly depended upon the composition of alloy and partial pressure of nitrogen. For instance, the addition of manganese, chromium, molybdenum and vanadium increased the solubility of nitrogen whereas the addition of carbon, silicon and copper decreased it [1,2]. The excellent nitrogen solubility was obtained by reducing the melting temperature and by increasing the nitrogen pressure. The solid solution hardening was greatly influenced by the nitrogen content. It was due to the misfit of the nitrogen atoms with the austenite crystal lattice. So this nature of N atom has the greatest effect on yield strength among other alloying elements. The atomic size of nitrogen was small when compared with that of iron, chromium and nickel making it as an interstitial element in steel [3]. In some cases the nitrogen was used as a replacement element for nickel in medical applications. Nickel can cause allergy for some users so replacing it with nitrogen not only increased the mechanical properties but also it was cost effective compared with other nickel composition alloys [4,5]. The nitrogen had a greater solubility in austenite than in ferrite; the solubility of nitrogen in austenite was 2.4% while in ferrite it was only 0.1% at the temperature of 1,100°C [6].

The nitrogen can be added to the alloy either in the form of nitrogenous alloying element or in gaseous state. The most common nitrogenous alloying elements included nitrided ferromanganese, nitrided ferrochrome, nitrided metallic manganese and vanadium-nitrogen compound [7]. In this study the nitrided ferrochrome was used as a nitrogenous alloying element. The five different weight percentage of nitrogen in steel such as 0.05%, 0.1%, 0.15%, 0.2% and 0.25% were chosen for the analysis. For every percentage increase of nitrogen the yield strength and hardness increased linearly. The tensile strength of 590 MPa was achieved in WC9 steel by nitrogen which was equivalent to the tensile property of C12A grade steel. While comparing in terms of cost, C12A grade steel was costlier than WC9-N grade steel, so WC9-N can be used as the replacement for C12A grade steel. The objective of adding nitrogen as an alloying element in WC9 steel was to meet the following requirements:

- Increase its mechanical properties so it can be used as the replacement for C12A grade low carbon steel which is of higher cost than the WC9 steel.
- Increase the tensile strength, without a reduction in ductility or fracture toughness.
- Increase the fatigue life and work hardening rate, the increasing percentage of nitrogen increases the hardness of WC9 steel linearly.
- Increase the ductile property of the component by increasing the value of percentage elongation and percentage reduction.
- Prevent fractures that were formed due to localized corrosion and to have a good surface finish.

* KONGU ENGINEERING COLLEGE, DEPARTMENT OF MECHANICAL ENGINEERING, PERUNDURAI, TAMILNADU, INDIA.

[#] Corresponding author; prabakr.p@gmail.com

2. Experimental details

2.1. Materials

The material chosen for the experimental procedure was WC9 grade steel which was a low carbon alloy steel. As per the American Society for Testing and Materials (ASTM) A217 standard, the chemical composition of WC9 grade steel is listed in the Table 1. According to the ASTM hand book the standard tensile, yield, elongation and reduction properties of WC9 steel is listed in the Table 2. The theoretical density of the grade was 7.82 g/cm^3. The major application of WC9 grade steel was making of valve bodies, pressure containing parts and in high temperature systems.

TABLE 1

Chemical composition of WC9 alloy

Element	Symbol	Wt. %
Carbon	C	0.05-0.18
Manganese	Mn	0.40-0.70
Phosphorous	P	0.04
Sulphur	S	0.045
Silicon	Si	0.6
Chromium	Cr	2.00-2.75
Molybdenum	Mo	0.90-1.20
Other residual elements	Cu, Ni, W	1.0

TABLE 2

Mechanical Properties of WC9 Steel from ASTM 217 handbook

Grade	Properties	Value
WC9	Tensile Strength [MPa]	485-655
	Yield Strength [MPa]	275
	Elongation in 2 in. [50 mm] min, %	20
	Reduction of Area, min, %	35

The nitrogen was used as the alloying element in WC9 steel because the addition of nitrogen increases the mechanical properties of the alloy linearly. The nitrogen was added to the alloy in solid form as nitride ferrochrome which had 60% Cr, 6% N and 0.1% C. At 0°C the density of nitrogen was 1.25 g/cm^3 which was very much less than the base alloy [7].

2.2. Preparation of WC9-N alloy

WC9 alloy with nitrogen concentrations of 0.05, 0.1, 0.15, 0.20 and 0.25% were prepared in an industrial induction melting furnace. The furnace was preheated at a temperature of 250°C. The iron scrap was then added to the crucible according to the weight percentage. All the other alloying elements including nitrided ferrochrome were added when the temperature of the substrate attains 700°C. The weights of the elements like chromium, silicon, manganese, molybdenum were measured on an electronic weighing scale for the required percentage to put into the furnace. In order to attain the homogeneity of the metal mixture, the substrate was allowed to melt upto 1590°C. Then the molten metal was tapped from the crucible to the ladle which was also preheated in order to eliminate the cold fusion of the molten metal.

After that the molten metal was poured into the mould which was already prepared using silica sand. The mould was prepared according to the shape of the test bar as shown in the Fig. 1. The total weight of the casting when it was poured into mould was 6 kg. The casting was allowed to solidify completely for 3 hours. After solidification, the casting was removed from the mould. Then the sample was heat treated by solution annealing process where it was maintained at 1140°C for 5 hours. Followed by water quenching in order to enhance the austenitic formation, the test bar was sent for final machining process. As like the above process five different samples of varying N% were prepared. The final machined WC9-N samples are shown in the Fig. 2. After all the machining process the samples were sent for microstructural study and then the mechanical properties are tested.

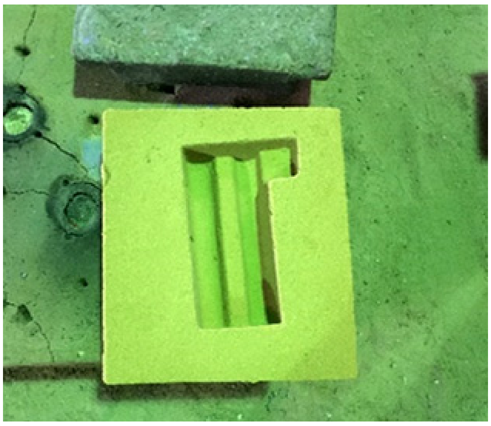

Fig. 1. Sand Casting Mould

Fig. 2. WC9-N samples

2.3. Specimen testing

First the composition of samples were analysed using the optical emission spectrometer (OES) which works in the principle of applying electrical energy in the form of spark generated between an electrode and a metal sample, whereby the vaporized atoms were brought to a high energy state within so-called "discharged plasma" [8]. Then the obtained composition was checked with that of the aimed values. After that the microstructure of WC9 alloy with different wt. % of nitrogen were examined by using an inverted metallurgical microscope (Getner Make). Before examining through the microscope the samples were initially rubbed with abrasive emery papers and then polished using the rotating disc cloth. The samples were then treated with an etching agent Nital of 3%.

The hardness test on the samples was carried out using a Rockwell hardness tester (TRS Model), in accordance with ASTM E18 standard at room temperature. A test load of 100 kgf was applied to the specimens. The diameter of steel ball indenter was 1.588 mm. The Rockwell hardness number (HRB) was noted for WC9-N alloy samples. An average of three readings was taken for each sample for hardness measurement.

The prepared samples were tested for tensile strength, yield strength, % elongation and % reduction. These tests were performed in a Universal Testing Machine (UTM) in accordance with the standard ASTM A370. The test specimen had the gauge diameter of 12.58 mm and gauge length of 50 mm. The experiments were conducted at room temperature. The load displacement curve, stress-strain graph were plotted with the obtained values to know the effect of nitrogen in the WC9 steel alloy.

3. Results and discussion

3.1. Microstructural study

The increase of heat treatment temperature and N percentage increased the austenitic stability thereby decreasing the ferritic phase. Nitrogen was a strong austenite former when it was heat treated at above 1100°C and the austenite content increased with the increase in nitrogen percentage and heat treatment temperature.

Fig. 3 shows the microstructures of samples (a), (b), (c), (d) and (e). From the microstructures, it was obvious that high solution annealing temperature results in an increase in austenite content. The Fig. 3a was the SEM image of the sample which had 0.05% of nitrogen, in which the ferrite formation was higher. But it was clear in the consecutive images that the ferrite content tends to decrease with increase in the N percentage. In Fig 3b, 3c, 3e the microstructure consisted of small amount of ferrite and bainite structure. The bainite was the transition phase between the ferrite and austenite, it commonly consists of cementite and dislocation rich ferrite. This high concentration of ferrite dislocation made it harder than the normal ferrite. In Fig 3d, the microstructure consists of martensitic structure.

3.2. Mechanical properties

The hardness test for various samples was taken using Rockwell hardness testing machine (TRS). The hardness values for varying N percentage in WC9 alloy was shown in the Table 3. From the Table 3 it was obvious that the hardness value increased at higher nitrogen percentage. The phase transformation to martensite was the main reason for the increase in hardness of the WC9-N alloy. At 0.25% of N the hardness value achieved was 37 HRC which was equivalent to Brinell hardness of 342 BHN which was more than the 158 BHN of standard WC9 alloy.

As discussed earlier the tensile property of the samples were carried out using the UTM machine. The variation of mechanical properties of WC9-N alloy with that of standard WC9 alloy samples were shown in the Table 3. From the table it was

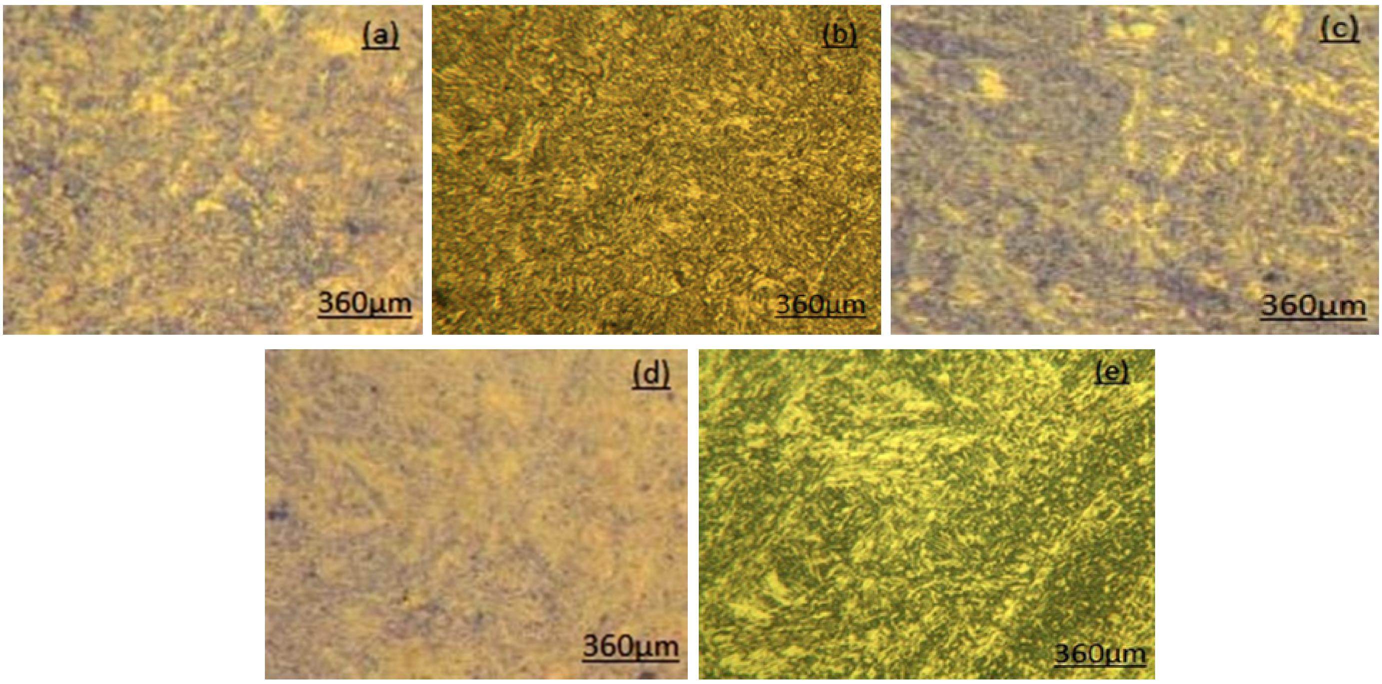

Fig. 3. SEM micrograph of WC9-N samples with wt. % of N at (a) 0.05, (b) 0.1, (c) 0.15, (d) 0.2 and (e) 0.25

TABLE 3

Mechanical properties

Elements	WC9	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Carbon C	0.1	0.1	0.1	0.1	0.1	0.1
Manganese (Mn)	0.5	0.5	0.5	0.5	0.5	0.5
Silicon (Si)	0.2	0.2	0.2	0.2	0.2	0.2
Sulphur (S)	0.005	0.005	0.005	0.005	0.005	0.005
Copper (Cu)	0.01	0.01	0.01	0.01	0.01	0.01
Phosphorus (P)	0.01	0.01	0.01	0.01	0.01	0.01
Chromium (Cr)	2.7	2.7	2.7	2.7	2.7	2.7
Nickel (N)	0.05	0.05	0.05	0.05	0.05	0.05
Molybdenum (Mo)	1.05	1.05	1.05	1.05	1.05	1.05
Nitrogen (N)	**0**	**0.05**	**0.1**	**0.15**	**0.2**	**0.25**
Tensile Stress (MPa)	486.55	489.88	496.77	531.4	538.92	592.35
Yield Stress (MPa)	356.44	468.54	475.65	493.67	531.68	563.21
Elongation %	26.88	27.68	28.09	28.48	28.92	30.23
Reduction %	44.04	42.33	50.17	52.59	54.92	56.22
Hardness (BHN)	158	195	228	236	255	342

observed that the increasing nitrogen percentage in WC9 alloy increased the tensile stress of the element.

The Fig. 4 shows the various values of tensile strength of WC9-N alloy with nitrogen percentage of 0.05, 0.1, 0.15, 0.20 and 0.25% respectively. As from the graph it was clear that the ultimate tensile strength of WC9-N alloy increased gradually with increasing percentage of nitrogen. Without the nitrogen the normal composition of WC9 alloy had the tensile stress of 486.55 MPa while increasing nitrogen percentage upto 0.25%, the tensile strength increased upto 592.35 MPa. Thus the higher tensile property was obtained in the WC9 alloy at a lower cost. There was an increase in the tensile property of the alloy because the nitrogen in the alloy occupies the interstitial position in the crystal lattice. Due to its smaller atomic size the nitrogen act as an interstitial element in steel making. As it occupies the interstitial space the density of the alloy increases thereby increasing the tensile property of the alloy.

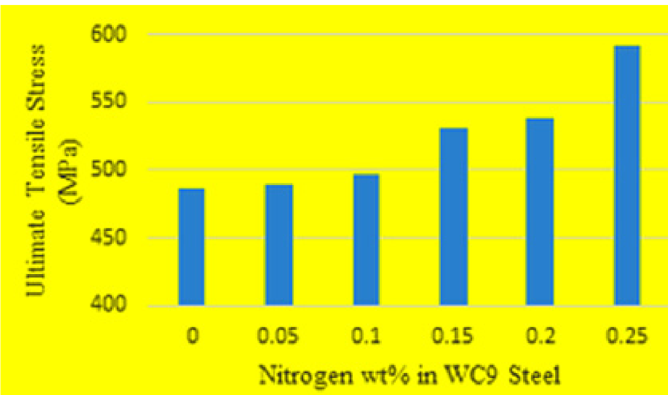

Fig. 4. Variation in tensile strength with wt. % of Nitrogen

The variation of yield strength of WC9 alloy with increasing nitrogen percentage was shown in Fig. 5. The yield strength was increased with the increase of nitrogen percentage. At 0.25% of N in WC9 alloy yield strength of 563.21 MPa was obtained which was 51.17% higher than the normal WC9 alloy which had a yield strength of 356.44 MPa. As we discussed earlier the dislocation of nitrogen atoms in the lattice was the main reason for increase in yield strength of the alloy.

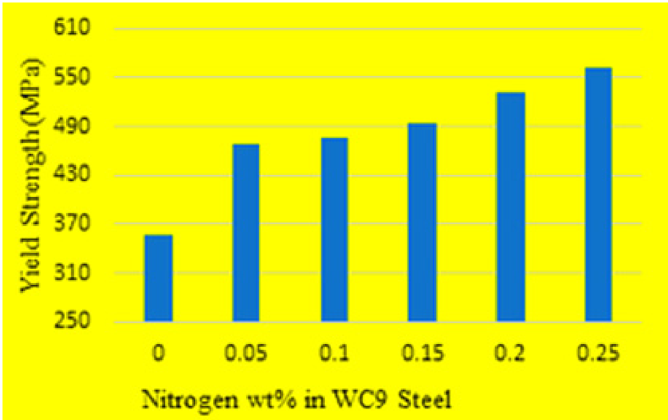

Fig. 5. Variation in yield strength with wt.% of Nitrogen

Fig. 6 is a graph showing the effect of nitrogen content on the percentage elongation (ductility) of the WC9-N alloy. The percentage elongation can be calculated using the formula shown in the Eq. (1). From Table 3 the percentage elongation was increased with increasing nitrogen percentage making the element more ductile than the standard WC9 alloy which had the elongation percentage of 26.88%. While the 0.25% of N in WC9 alloy has the 30.23% elongation. As the nitrogen occupies the interstitial position, it required more stress for elongation, thereby increasing the final gauge length of the test element. The final gauge length increased gradually from 0.05-0.25% increase of nitrogen, thereby making the element more ductile.

$$\text{Percentage Elongation} = \frac{\text{Final gauge length (in.)} + - \text{Initial gauge Length (in.)}}{\text{Initial gauge Length (in.)}} \times 100 \tag{1}$$

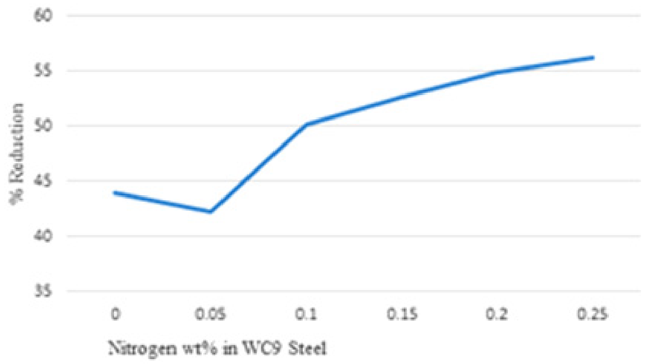

Fig. 6. Variation in % elongation with wt.% of Nitrogen

Fig. 7. Variation in % reduction with wt.% of Nitrogen

The percentage reduction of WC9-N alloy with increasing nitrogen percentage was plotted in the graph as shown in the Fig. 6. It was observed that the percentage reduction increased from 44.04% in standard WC9 alloy to 56.22% in WC9-N alloy where the percentage of nitrogen was 0.25%. The percentage reduction was calculated using the formula shown in the Eq. (2).

$$\text{Percentage Reduction} = \frac{\text{Area of original cross section} - \text{Minimum final area}}{\text{Area of original cross section}} \times 100 \quad (2)$$

3.3. Cost analysis

The obtained tensile strength at maximum N% in WC9 alloy was 592.35MPa. It was found to be equivalent to the tensile strength of C12A grade steel at its composition at lower end. So, WC9-N steel can be used as a replacement for C12A in its applications. The TABLE 4 shows the material cost for both the steel grades at the market value.

Total cost to produce 1kg of C12A grade steel = Rs. 65.49
Total cost to produce 1kg of WC9-N grade steel = Rs. 54.72
Difference in material cost between C12A and WC9-N = 65.49 – 54.72 = **Rs. 10.77**

From Table 4, it was clear that the material cost can be minimized by using WC9-N grade instead of C12A. Therefore, the profitability can be increased with minimal usage of alloying elements.

TABLE 4

Cost analysis between C12A and WC9-N alloy

S.No	Material	Cost per kg	C12A		WC9-N	
			Weight (kg)	Cost (Rs)	Weight (kg)	Cost (Rs)
1	MS Scrap	23	83.36	1917.3	93.68	2154.7
2	Coke	55	0.06	3.4	—	—
3	Fe Si	84	0.1	8.8	0.23	19.7
4	Lc Fe Cr	168	13.45	2259.8	0.78	131.2
5	Fe Mo	750	1.67	1250.4	1.66	1247.2
6	N2 Fe Cr	500	0.66	331.5	3.51	1756.6
7	Fe Si Zr	160	0.15	24	0.15	24
8	Ca Si	145	0.15	21.8	0.15	21.8
9	Al	70	0.05	3.5	0.1	7
10	Se	9700	0.02	194	0.01	116.4
11	Fe Nb	1730	0.13	229.4	—	—
12	Fe V	875	0.33	290.1	—	—
13	P Mn	170	0.23	38.6	—	—
14	HC Fe Mn	126	—	—	0.12	15.7
Total			**100.36**	**6572.6**	**100.39**	**5494.3**
Material cost per kg (Rs.)			—	**65.49**	—	**54.72**

4. Conclusion

In the present investigation, the influence of nitrogen alloying on WC9 low carbon steel, their microscopic structure and mechanical properties was investigated and it is observed that:

- The microstructural study shows the various phase transformation in WC9-N alloy. The increasing percentage of nitrogen makes the alloy in austenitic phase. As in the bainite phase the dislocation ferrite structure promotes the mechanical property.
- The ultimate tensile strength and yield strength of WC9-N alloy founds to be higher than that of WC9 alloy. The tensile strength and yield strength are tend to increase linearly with increasing nitrogen percentage. The tensile and yield strength of 592.35 and 563.21 MPa is obtained at nitrogen percentage of 0.25% in WC9-N alloy.
- The obtained mechanical properties of WC9-N alloy at 0.25% of N is equivalent to that of mechanical properties of C12A grade steel, at its composition at minimum end. While comparing the cost, the total material cost of C12A grade steel is 65.49 rupees while the material cost for WC9-N alloy is 54.72 rupees making it as a cost effective one.
- The hardness of WC9-N alloy at 0.25% of nitrogen is 342 BHN which is more than that of WC9 alloy, which has a hardness value of 158 BHN.
- The percentage elongation of WC9-N alloy is increased when compared to that of WC9 alloy. There is a 4% increase in elongation than standard WC9 alloy, thereby increasing the ductile nature of the element. Reduction percentage of WC9-N alloy also increases with increasing weight percentage of nitrogen.

Acknowledgements

The author would like to thank M/S. Vinayaka Electro Alloys (I) Pvt. Ltd. for invaluable assistance in this research.

REFERENCES

[1] S.K. Chung, C.R. Koch, A.F. Lynch, Flatness-based feedback control of an automotive solenoid valve, Ieee T. Contr. Syst. T. **15** (2), 394-401 (2007).

[2] J.R. Riba, A. Garcia, J. Cusidó, M. Delgado, Dynamic model for AC and DC contactors–Simulation and experimental validation, Simul. Model Pract. Th. **19** (9), 1918-1932 (2011).

[3] J.R.R. Ruiz, A.G.Espinosa, Design of an estimator of the kinematics of AC contactors. Int. T. Electr. Energy **19** (7), 933-948 (2009).

[4] Z. Wróblewski, A method of gamma-beta durability determination for reliability tests of brand-new AC electromagnetic contactors, Int. T. Electr. Energy **10** (4), 219-224 (2000).

[5] M.P. Paisios, C.G. Karagiannopoulos, P.D. Bourkas, Model for temperature estimation of dc-contactors with double-break main contacts, Simul. Model Pract. Th. **15** (5) 503-512 (2007).

[6] L. Jiang, Z. Li, G. Fan, L. Cao, D. Zhang, The use of flake powder metallurgy to produce carbon nanotube (CNT)/aluminum composites with a homogenous CNT distribution, Carbon **50** (5), 1993-1998 (2012).

[7] K. Liang, T.Y. Cheang, T. Wen, X. Xie, X. Zhou, Z.W. Zhao, C.C. Shen, N. Jiang, A. W.Xu, Facile preparation of porous Mn2SnO4/Sn/C composite cubes as high performance anode material for lithium-ion batteries. J. Phys. Chem. C **120** (7), 3669-3676 (2016).

[8] G. Wang, Y. Jianlei, J. Xueyan, Microstructure and mechanical properties of Ti–22Al–25Nb alloy fabricated by elemental powder metallurgy, Mat. Sci. Eng. A-Struct. **654**, 69-76 (2016).

[9] Y. Bai, X. He, Y. Li, C. Zhu, S.Zhang, Rapid synthesis of bulk Ti 2 AlC by self-propagating high temperature combustion synthesis with a pseudo-hot isostatic pressing process, J. Mater. Res. **24** (8), 2528-2535 (2009).

[10] C. Chen, L. Guo, J. Luo, J. Hao, Z. Guo, A.A. Volinsky, Aluminum powder size and microstructure effects on properties of boron nitride reinforced aluminum matrix composites fabricated by semi-solid powder metallurgy, Mat. Sci. Eng. A-Struct. **646**, 306-314 (2015).

[11] V. Ćosović, N. Talijan, D. Živković, D. Minićand, Ž. Živković, Comparison of properties of silver-metal oxide electrical contact materials, J. Min. Metall. B. **48** (1), 131-141(2012).