Improvement of tribological properties of Al alloy with CNTs and Nb nanopowder for industrial application

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Abstract. Unreinforced Al alloy lacks adequate tribological property required in service. To improve on this property, it must be reinforced with materials having good wear properties. Hence, Al was reinforced with CNTs and Nb and consolidated with spark plasma sintering (SPS). The wear experiment was conducted on a ball-on-disc Tribometer where loads of 10 N, 20 N and 30 N were systematically applied. The coefficients of friction (COF) of the sintered samples were subsequently obtained. The samples were weighed before and after each run to determine their weight losses which were used to compute their wear volumes. The experiment was designed and analyzed with Taguchi and ANOVA. Results showed that Al-8CNTs-8Nb composite had the best wear characteristics. Its COF got improved by 79% which shows that it is suitable for industrial applications.

1. Introduction
This study deals with improvement of Al alloy for industrial applications, especially in high transmission conductor. Ever since the conventional conductor, Aluminium conductor steel reinforced (ACSR) was found deficient in some operational areas, such as in operating temperature, coefficient of thermal expansion (CTE), corrosion, wear, and density [1], research has been on going to find a possible replacement. Al-CNTs-Nb composite is investigated in this study as a possible replacement for the conventional conductor. Traditionally, transmission conductors should be light in weight with high wear resistance, however, the base metal of this work, Al 1000 series alloy powder, is light in weight, corrosion resistant, electrically conductive, cost effective but poor in strength and tribology [2]. Therefore, Carbon nanotubes (CNTs) which have high strength, high thermal conductivity, high wear resistance and light weight [3]; Nb is a superconductor, a grain refiner, a fracture toughening additive and a wear resistant additive [4]. These properties should be able to improve the properties of Al alloy for industrial application. The fabrication process is spark plasma sintering (SPS) which is a non-conventional sintering method that utilizes high heating and cooling rates to minimize grain growth. The presence of plasma heating in SPS helps to vaporize impurities. More so, the low sintering temperature is necessary for energy conservation. And also, the short sintering time makes the process cost effective; while the simultaneous heating with application of pressure helps to achieve a solid compact at a temperature lower than the melting point of the material [5]. Even though SPS is a bulk synthesising technique, it is believed to be a surface improving process as it generates products...
with wear and corrosion resistance. Tokita [6] produced high wear resistance nozzle with SPS. Many researchers have worked on improving the properties of Al alloy with ceramics, particulates, metal powders and even thermo-mechanical treatments. For instance, Ehsan et al [7] reinforced Al alloy with TiC, Loto and Babalola [8] improved Al with SiC using SPS. Zawadzki et al [9] improved AA7075 alloy with heat treatment and shot penning, and Kyziol et al [10] surface-modified AA2024 by chemical vapour deposition. However, SPS of Al-CNTs-Nb composite aimed at improving the wear characteristics of Al alloy has not been reported anywhere in the literature despite the excellent properties of these reinforcements. This study therefore, is aimed at utilizing the characteristics of the CNTs and Nb additives in one hand and SPS on the other hand to improve the tribological properties of Al alloy for use in transmission conductor.

2. Material and methods

2.1. Material

Aluminium alloy powder (1000 series) of purity (99.9 wt.%) with particle size of 40 nm; multi-walled carbon nanotubes of purity (99.5 wt.%), diameter (10-30 nm), (length 5-20 µm); and Niobium nano powder of purity (99.9 wt.%) and particle size of 20-40 nm, which were supplied by a company in China named Hongwu International Group, constituted the materials used in this study. Masses of the powders required to reflect the Taguchi design of experiment (DOE) in table 1(a) were weighed out and mixed. The CNTs with Young’s modulus of 950 -1500 GPa, Poisson ratio of 0.32, thermal conductivity of 3000 W/mK and produced by chemical catalytic vapour deposition (CCVD) were initially agitated to de-agglomerate particles with a Tubular Shaker Mixer at an angular velocity of 69 r/min for 8 h with steel balls incorporated therein. Thereafter, the whole powders were blended together at 110 rpm for 10 hours with balls made of high carbon steel at a ball to powder (B:P) ratio of 2:5 following Chieh et al, Ujah et al [11, 12].

Table 1. (a) Taguchi DOE (L9 3² Orthogonal array) for sintering. (b) Taguchi DOE (L9 3³ Orthogonal array) for tribology experiment.

| Exp no | Al (wt.%) | CNTs (wt%) | Nb (wt%) | Description       | Load (N) |
|--------|-----------|------------|----------|-------------------|----------|
| 1      | 98        | 1          | 1        | Al-1CNTs-1Nb      | 1        |
| 2      | 95        | 1          | 4        | Al-1CNTs-4Nb      | 2        |
| 3      | 91        | 1          | 8        | Al-1CNTs-8Nb      | 3        |
| 4      | 95        | 4          | 1        | Al-4CNTs-1Nb      | 4        |
| 5      | 92        | 4          | 4        | Al-4CNTs-4Nb      | 5        |
| 6      | 88        | 4          | 8        | Al-4CNTs-8Nb      | 6        |
| 7      | 91        | 8          | 1        | Al-8CNTs-1Nb      | 7        |
| 8      | 88        | 8          | 4        | Al-8CNTs-4Nb      | 8        |
| 9      | 84        | 8          | 8        | Al-8CNTs-8Nb      | 9        |

2.2. SPS technology

Optimized SPS parameters reported elsewhere [12] was used to sinter the samples. They include 630°C 30 MPa, 10 mins and 200°C/mins. The blended powders were singly sintered one after the other with these parameters at a vacuum of 0.605e-3 bar, relative pressure of -500e-3 bar, and absolute pressure of 1.2e-3 bar. Graphite paper was used to fix at the wall of the die to aid in homogenous heating and for easy removal of the sample after sintering. The number of samples produced was 10 and they were cut for analysis. Figure 1 shows an illustration of SPS mechanism and sintered sample. It can be seen that at the start of sintering (figure 1(a)), heat is circulated within the powder mass through plasma heating. But once neck formation takes place, heat is transferred to the adjacent particles via joule heating.
2.3. Tribology test

The 10 samples were prepared for wear test by cutting them into (20 x 20 x 2 mm) sizes followed by metallographical polishing. The test was conducted at room temperature. The Tribometer consists of ball-on-disc reciprocating machine capable of generating the coefficient of friction (COF) of test samples. The ball is made of Grade 25 alloy steel E52100, 6.35 mm in diameter. The stroke length was 5 mm, velocity was 3 mm/s, time was 360 seconds and the loads were 10 N, 20 N, and 30 N. The masses of the sample before and after each test were taken and used to compute the wear volume as shown below:

\[
\text{Wear volume (cm}^3) = \frac{(M_1 - M_2)}{\rho}
\]

Where \(M_1\) and \(M_2\) [g] are masses obtained before and after each wear tests respectively, \(\rho\) [g/cm\(^3\)] is the actual density of the composite material. The tribology test was analyzed with statistics by firstly designing the experiment with Taguchi DOE (L9 3\(^3\) Orthogonal array) and analyzing it with ANOVA. The Taguchi DOE is shown in table 1(b).

3. Results and discussion

The TEM micrograph of the start-up powders are shown in figure 2. It can be seen that the Al particles (20 -100 nm) are spherical in shape (figure 2(a)), CNTs are made of cylindrical tubes entangled to one another (figure 2(b)) and Nb particles (10 – 80 nm) are spherical too with some tiny particles clinging to bigger ones to form some sorts of agglomeration, though may be due to packaging from the industry. The agglomeration of CNTs was due to its high aspect ratio [13].

Scanning electron microscope (SEM) images of sintered samples are shown in figure 3. It can be seen that there was grain refinement, homogenous dispersion of reinforcements and cohesive matrix/reinforcement interface except for figure 3(c) which has pores. The pores would definitely
affect the sample as could be seen later on.

Figure 4 shows coefficient of friction (COF) and wear volume profiles. It can be seen that the COF decreased as the load increased (figure 4(a)). This could be because higher loads increased the temperature of the surface and subsequently softened it thereby reducing the COF [14]. However, Wang et al [15] had a contrary observation where it was seen that COF increased with increase in applied load. The COF of pure Al was higher than that of the reinforced composites and got improved by 79% (from 0.48 for pure Al to 0.10 for Al-8CNTs-8Nb). The improvement can be attributed to the incorporated reinforcements which induced refinement of the grain sizes as can be seen in figure 3(d), well dispersed reinforcements and compact grain boundaries [4-7]. Figure 4(b) shows wear volume of the sintered samples subjected to wear test. Here it could be seen that unreinforced Al alloy had the highest wear volume while Al-8CNTs-8Nb had the least wear volume. The reduced wear volume in the composites was because the reinforcements had high wear resistance and the SPS processing technique impacted positively on the products [5,6,14]. Also observed was that the higher the load, the higher the wear volume which could be attributed to the fact that the increased load increased the shearing force on the surface thereby removing more materials from the surface [14,15]. The wear volume got improved by 23% (from 12.8 ± 1.2 µm³ for Pure Al alloy to 9.8 ± 2.0 µm³ for Al-8CNTs-Nb composite).

![Figure 4. Tribology profiles (a) COF (b) wear volumes.](image)

![Figure 5. Optical microscopy of some worn surfaces.](image)

Figure 5 shows the optical micrographs of worn surfaces. It can be seen that unreinforced pure Al alloy had more severe wear track than other samples (Figure 5(a)). In Figure 5(d), the wear track was very shallow which concur to the low COF and low wear volume of Al-8CNTs-8Nb composite. Figure 5(b) shows wear track that is severe though less than that of pure Al. In Figure 5(c), the wear track still reduced. The trend shows that the higher the quantity of reinforcement, the shallower the wear tracks
which could be interpreted from the angle that the protective films generated by the reinforcements increased with their weight fractions. The wear mode observed in the whole samples was that of abrasion. It was noticed also that the higher the load, the more severe the wear track. Figure 6 shows coefficient of friction (COF) profiles of Al-CNTs-Nb at 20 N loading over time. The outstanding feature of the profile is that there are two main stages, the running-in stage and the steady state stage. The friction rises rapidly from zero mark to the maximum value, and then falls down before it stabilizes.

Figure 7 shows Taguchi-ANOVA analysis of tribology result. In figure 7(a), it could be seen that load 1 (10 N) had the highest influence on the outcome of the coefficient of friction. This was confirmed in figure 7(b) where load had the highest percentage contribution of 67.12% to the outcome of the tribology experiment. Next in significance was Nb reinforcement (level 3, that is 8 wt.%) and then CNTs (level 3, that is 8 wt.%). The interpretation of this is that 8 wt% of both Nb and CNTs reinforcements produced highest effect on the reduction of COF of the composite. This was confirmed in figures 7(c) & 7(d) where 10 N load, 8 wt.% Nb and 8 wt.%CNTs gave the highest mean values and the least signal-to-noise ratios respectively. Hence, Al-8CNTs-8Nb has shown the least COF in this analysis.

4. Conclusion

The experiment has been conducted and findings show that wear resistance of Al composite improved by 79% in terms of COF and 23% in terms of wear volume. The sample with the best wear characteristics was Al-8CNTs-8Nb with a density of 2.72 g/cm³. Taguchi and ANOVA analyses concurred with this result. These improvements could be attributed to the generation carbon film by CNTs which acted as a solid lubricant and the Nb₂O₅ which was formed by Nb that acted as a protective layer against wear, together with the SPS route which aided in improving the properties of the composite so far developed. So, the properties achieved concur that Al-CNTs-Nb composite is a robust material for industrial application such as in high power transmission conductor and its core.

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