Tribological properties of aluminium reinforced by TiCN nanosized powder

M Kolev\textsuperscript{1,3}, S Valkov\textsuperscript{2}, R Lazarova\textsuperscript{1}, P Petrov\textsuperscript{2}, R Dimitrova\textsuperscript{1} and V Dyakova\textsuperscript{1}

\textsuperscript{1}Acad. A. Balevski Institute of Metal Science, Equipment and Technology with Hydro and Aerodynamic Center, Bulgarian Academy of Sciences, 67 Shipchenski Prohod Blvd., 1574 Sofia, Bulgaria
\textsuperscript{2}Acad. E. Djakov Institute of Electronics, Bulgarian Academy of Sciences, 72 Tsarigradsko Chaussee, 1784 Sofia, Bulgaria

E-mail: mihail1kolev@gmail.com

Abstract. The work is aimed at investigating the tribological properties of aluminium surface modified by electron beam with TiCN nanoparticles as a reinforcing phase. In the course of study, the microstructure is observed and the surface of treated and untreated specimens are tested and compared in order to determine the influence of the reinforcing TiCN phase on the coefficient of friction, the friction force, and the linear mass wear. The tribological properties are evaluated by the pin-on-disk technique in a lubricating medium. The TiCN nanoparticles embedded in the aluminium matrix are obstacles to any dislocation motion, which improves the tribological properties of the samples’ surfaces. The acting wear mechanisms are discussed.

1. Introduction

Nanocomposites have been the subject of research and development over the past two decades. They proved to be suitable alternatives in overcoming the limitations of micro-composites and of monolithic samples. When the size of a particle is less than a certain level, changes are observed in its properties \cite{1}. Furthermore, as the dimensions reach the nanometer scale, large alterations in the interactions at interfaces between phases take place, an important phenomenon that could be used as a way of enhancing a given material’s properties \cite{2}. Improved mechanical properties have been observed by reducing the size of ceramic particles from a micrometer to nanometer level, which also allows for the use of lower volume fractions of nanoparticles \cite{3-4}.

Controlling the size of the nanoparticles helps to achieve the desired wavelength region for intended applications, as windshields, light reflection and scattering, clear glasses etc. \cite{5-6}.

Aluminium matrix nanocomposites are of crucial importance in the progress and the manufacture of advanced materials and methods in the automotive, aerospace and transportation industries \cite{7-12, 13, 14, and 15}.

In some cases, the surface of the components must be of high strength, hardness and wear resistance, while the core should retain high plasticity and toughness, as for example in engines pistons. In these cases, producing a surface aluminium matrix nanocomposite (SAMNs) proved to be an efficient way of improving the surface mechanical properties of the components. For this purpose, various techniques are being used: high-energy laser-beam treatment, plasma spraying, cast sintering

\textsuperscript{3}To whom any correspondence should be addressed.
and electron-beam irradiation. In this work, we used TiCN nanoparticles as a reinforcing phase and electron-beam treatment as a method for incorporating them.

Our previous research has proved the high microhardness of surface layers obtained by coating the surface of an aluminium substrate with a mixture containing TiCN nanoparticles and subsequently irradiating the surface by an electron beam in a circular pattern [16, 17]. Furthermore, the nanocomposites exhibit a reduced wear loss compared to the same alloys without a nano-reinforcement phase [18-21].

In the present paper, the effect is studied and compared of TiCN nanoparticles as a reinforcing phase on the coefficient of friction (COF), the friction force and the mass wear of specimen with and without surface coating. We expected that the wear resistance would be significantly increased, given that TiCN is a reinforcement for aluminium and its alloys and possesses superior mechanical properties, such as high hardness (HV 2500-3000), high toughness and excellent wear resistance, even compared with other ceramic particles, as SiC, TiC, TiN, TiB₂ and B₄C [2].

2. Materials and methods

Three cylindrical specimens made of aluminium of technical purity (99.5% Al and 0.5% other elements) were tested under frictional lubrication conditions. The dimensions of the samples were 12 mm in diameter and 10 mm in height. The first sample was of untreated Al and marked 1-Al. The second was of Al subjected to electron-beam treatment (EBT), marked 2-Al, and the third, of Al coated with a mixture containing TiCN nanoparticles and subjected to surface EBT – 3-Al.

The treated samples were prepared following the method used in [17]. Figure 1 shows the three experimental specimens before being subjected to tribological tests.

2.1 Metallography

Cross-sections perpendicular of the flat surface were prepared for microstructure analysis. They were wet-ground by grinding paper up to 4000, mechanically polished and etched by a 0.5-% aqueous solution of HF. The observations were carried out by a PolyvarMet metallographic microscope at magnifications up to 1000×. The microhardness was measured by a MicroDuromat 4000 device, with a load of 20 g, time of reaching the load of 10 s and holding time of 10 s.

2.2 Tribology

The “pin/ball on the disk” tests were carried out on a Ducom Instruments installation certified by the American Society of Tribology (figure 2).

The tribological installation shown in figure 2 functions as follows: The cylindrical sample is positioned in the holder which is located at one end of the loading beam. The test surface of the sample is in direct contact with the surface of a counterpart disk, whose hardness is 60 HRc. The counterpart is mounted on a special holder designed to conduct a lubricant test. The drive is powered by an electric motor rotating at a constant speed; for the purpose of our tests, we chose 65 rpm. At the opposite end of the loading beam, there is a static working load of 110 N. The installation has two sensors for precise measurement of the friction force. The results are plotted in real time using specialized software that calculates the friction coefficient after the test is completed. The three tests reported here were conducted under
frictional conditions with 50 ml of 10W40 motor semisynthetic oil as a lubricant, with the contact surface of the specimens submerged in the medium.

The mass wear was determined by a Boeco BAS32 Plus electronic balance with a scale accuracy of 0.1 mg and the possibility of automatic internal calibration.

The methodology for the tribological testing consists of the following steps:

- **Preparation of the experimental specimens**
  The specimens had the following dimensions: diameter 12 mm and height 10 mm. Their contact surfaces were wet-ground by sandpaper size 2500.

- **Mass wear determination**
  Before each measurement, the samples were cleaned and degreased to remove organic and mechanical particles and dried by ethyl alcohol to prevent electrostatic effects. The samples were measured before and after the test and the difference in their masses was established.

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m = m_0 - m_f \text{ [mg]},
\]

\( m \) being the difference between the initial \((m_0, \text{before friction})\) and final \((m_f, \text{after end of test})\) masses.

**Experimental conditions:**
- Room temperature of 22 °C;
- After determining the initial mass prior to the tests, the specimens are mounted on the installation holder and positioned at a diameter of 110 mm on the contact surface of the abrasive body;
- The contact surface of the test body is immersed in 10W40 engine-semi-synthetic motor oil for two hours;
- The distance covered by each sample is 2700 m;
- The speed of rotation the counter disk 65 rpm;
- The stationary load applied is 110 N.

- **Friction force measurement**
  The friction force registered by the sensor is plotted in real time as friction force (N) vs time (s) and friction force (N) vs distance (m).

- **Friction coefficient measurement**
  The friction coefficient is calculated by the software after the end of the test and the plots friction coefficient vs time (s) and friction coefficient vs distance (m) are constructed.

### 3. Results and discussions

#### 3.1 Microstructure and microhardness

The matrix of the aluminium samples is an α- solid solution with coarse grains. The microhardness values near the edge and in the core of sample 1-Al are the same – 29 kg/mm². A layer of about 20 –25 μm is formed at the surface after electron-beam treatment (figure 3 a). The microhardness of the layer of sample 2-Al is 78 kg/mm²; in its core it is 29 – 30 kg/mm². This means the EBT itself hardens the material by a factor of 2.7.

The layer in sample 3-Al is much harder (814 kg/mm²) than the matrix (38 kg/mm²), or by a factor of 21.4 (figure 3 a). This is due to the hard TiCN particles incorporated in the layer as we have found previously [17]. The nanoparticles, whose diameter is commensurate with the dislocations, represent obstacles to the motion of dislocations. The Orowan mechanism is the main strengthening mechanism acting in aluminium nanocomposites [22, 23]. The strengthening effect is the result of the formation of multiple dislocations (Orowan loops) around the reinforcement particles, as argued by many

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**Figure 3.** Sample 3-Al: a) LM and b) TEM image showing the nanoparticles in the Al matrix.
researchers [24-26]. This could serve as a prerequisite for increasing the hardness, the strength properties and the wear-resistance.

3.2 Tribological properties

The data obtained for the mass wear for the entire experimental cycle are presented in table 1 and figure 4. The untreated sample lost more mass compared to the other two samples, namely, 1-Al reduced its mass due to surface friction by 0.0109 g more than the electron-beam treated sample 2-Al, and 0.0524 g more than the sample 3-Al with a surface coated with nanoparticles and electron-beam treated. Furthermore, there is a significant mass-wear difference between the surface-treated samples: the electron-beam treated sample 2-Al lost 0.0415 g more mass than sample 3-Al, which was coated with nanoparticles and electron-beam treated. The hard nanoparticles dispersed in the soft aluminium matrix present obstacles that are difficult overcome, thus hindering the motion of dislocations. As seen in figure 3b, the dislocations create a type of network, or meshes of sub-grains boundaries around the nanoparticles. This could explain the higher wear resistance of the layers containing TiCN nanoparticles in comparison with the layers without such embedded particles. Table 2 summarizes the mean experimental values for the friction force and the friction coefficient obtained at time/distance of 83 min/1800 m for each of the test samples at a load of 110 N.

As seen in figures 5 and 6, after 2000 s of elapsed time and, respectively, 800 m of distance passed, the surfaces of the sample and the wear disk reach a good working contact. One can also see (figure 5) that the friction force for sample 1-Al is greater by 5 N than that for 2-Al and by 8 N greater than that for 3-Al. A similar behavior is observed in figure 6 comparing the friction coefficient of each of the

Table 1. Experimental results of mass wear of the samples at test time/distance of 160 min/2700 m.

| Sample | Initial mass, g | Mass after friction, g | Mass wear, g |
|--------|-----------------|------------------------|--------------|
| 1-Al   | 1.7829          | 1.7290                 | 0.0539       |
| 2-Al   | 2.9264          | 2.8833                 | 0.0430       |
| 3-Al   | 3.1795          | 3.1778                 | 0.0015       |

Table 2. Average friction force and friction coefficient of test specimens at test time/distance 83 min/1800 m.

| Sample | Friction force, N | Coefficient of friction |
|--------|-------------------|-------------------------|
| 1-Al   | 13                | 0.11                    |
| 2-Al   | 8                 | 0.07                    |
| 3-Al   | 5                 | 0.05                    |
test specimens. Sample 1-Al has the highest coefficient of friction 0.11, and sample 3-Al, the lowest one – 0.05.

The values quoted above of the coefficient of friction are mainly in the range from 0.1 to 0.05 due to the composites with added nanosized reinforcement generally having a lower coefficient of friction and a higher wear resistance than alloys with unreinforced matrices [18].

The wear resistance of aluminium reinforced with 4.5% TiO₂ is improved by about a factor of two compared to that of Al for loads in the interval 5 – 25 N [27].

Embedding TiCN nanoparticles in an aluminium matrix increases the resistance of the material to friction because of the nanoparticles’ high hardness and their commensurability with the dislocations’ dimensions; they thus act as obstacles to the motion of the dislocations and the material becomes stronger.

4. Conclusions
The analyses performed allow us to draw the following conclusions:
• The electron-beam treatment of the surface of aluminium samples causes the formation of a layer; namely, a surface zone where TiCN nanoparticles are incorporated. Their presence determines the increase in the microhardness.
• The tribological characteristics – mass wear, friction force and coefficient of friction, of the samples with nanocomposite layer are improved compared to those of the samples without such a layer when both are subjected to EBT.
• The increased microhardness and improved tribological characteristics are due to the higher microhardness and better tribological characteristics of TiCN nanoparticles in comparison of Al-matrix and the obstruction to the dislocations motion created by the nanoparticles in the Al matrix.

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