Materials and Design 32 (2011) 3865–3875

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Wear improvement of sol–gel silica coatings on A380/SiCp aluminium composite substrate by diode laser sintering

A.J. López *, A. Ureña, J. Rams

Dept. de Ciencia e Ingeniería de Materiales, ESCET, Universidad Rey Juan Carlos, C/ Tulipán s/n, Móstoles 28933 Madrid, Spain

ARTICLE INFO

Article history:
Received 4 January 2011
Accepted 2 March 2011
Available online 5 March 2011

Keywords:
A. Glasses
C. Rapid solidification
E. Wear

ABSTRACT

In this investigation, a high power diode laser (HPDL) was used to induce the microstructural refinement on the surface of a SiC particulate (SiCp) reinforced Al-based metal matrix composite (A380/SiC/20p) and, at the same time, to the sintering of a sol–gel ceramic layer deposited on the surface of the mentioned substrate. The purely inorganic silica ceramic coating was synthesised through the organic sol–gel route, using tetraethoxysilane (TEOS) as alkoxide precursor and the dip-coating as the deposition technique on the surface of the A380/SiC/20p composite. Optimisation of the laser parameters led to homogeneous and free of cracking coatings and also to the refinement of the surface microstructure of the substrate by means of the dissolution of the intermetallic precipitates, the decrease in the aluminium dendrites size and a better distribution of the silicon carbide particles. Unlubricated pin-on-disc wear tests confirmed the increase (89% in terms of specific wear rate drop) in the wear resistance of the coated substrates treated by the HPDL.

1. Introduction

The aluminium matrix composites reinforced with silicon carbide particles (SiCp) have high wear resistance, stiffness and thermal stability. These properties make them suitable for a number of applications in automotive, aerospace and engineering components [1]. Most of these composites present low corrosion properties, because of the presence of discontinuities in the natural alumina protective coating at the interfaces between matrix and reinforcement, as well as because of the existence of coarse intermetallic phases at their surface [2].

In order to improve the corrosion behaviour of different composites and alloys, without reducing their tribological properties, ceramic coatings are usually applied [3,4]. These coatings can be obtained by Physical Vapour Deposition (PVD) and Chemical Vapour Deposition (CVD) process; however these technologies demand cost intensive equipment. Sol–gel processing is a cost-effective alternative to generate ceramic coatings with the required thickness for many purposes, including wear and corrosion protection.

Authors have studied in previous works the protection of aluminium matrix composites by means of silica coatings through sol–gel methods [5] to increase their corrosion [3] and wear resistance [6]. Moreover, alternative method was studied in order to recover the mechanical properties of the coated composite based on quenching of the coated substrate after the heat treatment of densification [7]. In these previous works densification of the final ceramic coatings were achieved by heat treatments in oven.

Nowadays, different densification processes for the fabrication of ceramic sol–gel coatings on metallic substrates are under investigation, in order to enhance the coating consolidation and to develop time-saving techniques, such as densification by ultraviolet irradiation and water vapour [8], high power CO_2 and Nd:YAG lasers [9,10], or excimer laser annealing [11]. Even, a novel laser technique has been used for the in situ deposition of the ceramic sol–gel layer by immersing the substrate in the sol solution, an irradiating its surface with a laser while it was still inside the sol [9]. In previous investigation [7] authors have demonstrated the effectiveness in corrosion protection of aluminium matrix composites by means of silica sol–gel coatings sintered using a high power diode laser (HPDL).

Pardo et al. have successfully refined the surface microstructure of different aluminium matrix composites, reinforced with different volumetric proportion of SiC particles, via HPDL [12,13]. The laser surface melting (LSM) reached allows, after the optimisation of the laser parameters, not only the dissolution of the cathodic second phase particles (intermetallic precipitates) and therefore increases the corrosion behaviour of the composite, but also a refinement of the dendrite size and a better distribution of the SiC particles (SiCp) with the subsequent enhancement of the surface hardness.

The aims of the present work are to induce the microstructural refinement on the surface of a SiC particulate reinforced Al-based metal matrix composite (A380/SiC/20p) and the sintering of a silica

* Corresponding author. Tel.: +34 914888083; fax: +34 914888150.
E-mail address: antoniojulio.lopez@urjc.es (A.J. López).

0261-3069/$ - see front matter © 2011 Elsevier Ltd. All rights reserved.
doi:10.1016/j.matdes.2011.03.004

sol–gel ceramic layer deposited on the surface of the mentioned substrate. For this double aim a HPDL was used. The technique resulted to be suitable for both aims after the optimisation of its parameters. The influence of this laser parameters in the final ceramic coating densification and tribological properties were studied. Wear behaviour of the annealed ceramic layers were investigated using the pin-on-disc test under unlubricated and room temperature conditions, and observing the worn tracks and the debris particles formed during the wear test. Discussion of the wear results were made taking into consideration the microstructural changes in the surface of the composite substrate and the densification of the ceramic sol–gel layer.

2. Experimental method

2.1. Substrates

Coatings were made on $35 \times 20 \times 2$ mm^3 cast aluminium composite samples reinforced with 20 vol% SiC in form of polyhedrical particles (A380/SiC/20p). Nominal composition of the aluminium matrix alloy (in wt%) is: 10.07 Si, 0.97 Fe, 3.13 Cu, 0.59 Mn, 0.39 Mg, 0.01 Zn, 0.08 Ti, traces of Cr and Sr, and balance of Al. All samples were ground to P800 finish with SiC grit papers before being coated.

2.2. Sol–gel coating preparation

Silica layers were fabricated from a pre-hydrolysed solution of the silica precursor using the sol–gel route. A homogeneous, clear and transparent solution was prepared by dissolving tetraethoxysilane (TEOS) in ethanol. The molar ratio was TEOS/C_2H_5OH = 1/11. This solution was only partially hydrolysed by adding 0.1 M HCl acidulated water (molar ratio TEOS/H_2O = 1). This mixture was magnetically stirred for 2 h without heating and aged at room temperature for 30 min more. This procedure was the one that provides optimal coatings, avoiding the appearance of cracks on the sample surface, as it has been shown before [5–7].

Single layer coatings were obtained by dipping aluminium matrix composite samples on the pre-hydrolysed solution and extracting them at a controlled speed of 35 cm/min. Then, the coatings were dried in an oven at 100 °C for 1 h to eliminate the solvent excess and to promote the gelation process [12,14,18].

2.3. Coatings densification

After drying the coatings, some coated samples were treated at 500 °C for 1 h in a pre-heated electrical oven and cooled inside it to room temperature (conventional heat treatment). These samples were named as "HT" and were fabricated by the conventional sintering of the sol–gel coatings [5,7,14] as comparative purposes with that obtained by laser treatment.

Other set of samples were treated with a High Power Diode Laser (HPDL) of GaAsAl from ROFIN–DILAS (model DL13S) with a maximum power of 1300 W and with an emission wavelength of 940 nm. The laser radiation allowed the evaporation of the organic remnants from solution and the subsequent sintering of the coating [12].

Control of the power density of the laser radiation was achieved by placing the laser head on an anthropomorphic robot from ABB. This procedure, previously used [12], also allowed controlling the scan speed and the distance between consecutive laser lines. The area of the rectangular laser spot was kept at 5 mm^2 and the incidence angle used was 5° to normal. The laser parameters used for coating sintering are indicated in Table 1, and the input energy used during the laser process is also estimated as a function of scan speed according to the following equation

$$\mathrm{INE(J/m)} = \text{input laser power(W)/scan speed(mm/s)} \quad (1)$$

Table 1
Laser parameters used for silica sol–gel densification and input energy of the laser processes.

Condition	Power (W)	Speed (mm/s)	Interlinear distance (mm)	Input energy (kJ/m)
DL1	950	80	1.2	11.9
DL2	950	60	1.2	15.8
DL3	950	50	1.2	19.0
DL4	800	50	0.6	17

2.4. Substrate and coating characterization

The microstructure of the substrate in the as-received state, and after being coated as well as coatings morphology was characterized by Scanning Electron Microscope (SEM) in a Hitachi S-3400 N at high vacuum conditions, and by Energy Dispersive X-ray Spectrometry (EDX). Platinum metallization by Sputtering was necessary before the SEM characterization of the sol–gel coatings because of the ceramic nature of the layer.

Hardness of the as-received substrate and after heat and laser treatments was evaluated on the top surface by means of Vickers hardness tester (Testor 2100 from Instron) using 0.5 N load to determine the mechanical response, in terms of Vickers hardness number (HV) of the substrate-coating system after the different processes of coating consolidation applied. The values shown are the average of ten different measurements.

2.5. Wear tests and characterization of worn tracks

Wear properties of the coatings were investigated using a pin-on-disc tribometer (MT/10/SCM from MICROTEST) under unlubricated conditions at room temperature. The counterbody was a 6 mm diameter steel ball. The tests were carried out with 1 N load and a rotating speed of 0.1 m/s for a total wear distance of 150 m. Mass loss was measured with a 10^{-5} g analytical balance before and after the tests. In order to take the repeatability into account, the results of the wear behaviour were obtained from the average of four tests on each material in the steady-state of sliding.

Volume loss after the wear test was determined in each specimen from the mass loss measurements considering the real density of the coating. From it, the specific wear rate (mm^3/N m), as defined by Friedrich et al. [15–17], was calculated dividing by the normal applied load and the total wear length.

SEM and Energy Dispersive X-ray Spectrometry (EDX) were used to analyse the morphology and chemical structure of worn surfaces and wear debris formed after the pin-on-disc tests.

3. Results

3.1. Microstructure and hardness of treated substrates

The microstructure of the untreated A380/SiC/20p composite reveals large aluminium α–Al dendrites, with geometrically-shaped SiC particles and large and split AlFeSiMn–α intermetallic phases placed at interdendritical positions (Fig. 1a). In addition, Al_2Cu(Mg) and AlCuFe(NiMg) intermetallic phases were also present in the microstructure in intracrystalline places [18–20].

The substrate microstructure was not modified during the heat treatment applied in an oven for the densification of the coating, as it is shown in the transversal section of the composite (Fig. 1b), but