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Ta_2AlC and Cr_2AlC Ag-based composites—New solid lubricant materials for use over a wide temperature range against Ni-based superalloys and alumina

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Abstract

The tribological performances of the two new composite materials, consisting of the layered ternary carbides (MAX phases), Ta_2AlC or Cr_2AlC, and 20 vol.% Ag, were investigated in the temperature range from ambient to 550 °C against a Ni-based superalloy, SA (Inconel 718) and alumina counterparts. Over the entire temperature range, the wear rates, WRs, during dry sliding against the SA counterparts for the Ta_2AlC–Ag and Cr_2AlC–Ag composites were $<5 \times 10^{-5}$ and $<10^{-4}$ mm^3/Nm, respectively. The friction coefficients, μ, were <0.5. The WRs of the SA counterparts were also relatively low ($<10^{-4}$ mm^3/Nm). Under thermocycling conditions, the tribological performance of the MAX/Ag composites-Inc718 tribocouples got better with sliding distance. When the composites were tested against Al_2O_3, their WRs at moderate temperatures were also $\approx 10^{-5}$ mm^3/Nm, but at 550 °C the WRs increased by about an order or magnitude. Both composites had tensile strengths, σ_t, >150 MPa, compressive strengths, σ_c, >1.5 GPa at ambient temperature and σ_t > 100 MPa at 550 °C. Their good tribological performance together with decent mechanical properties and machinability render them promising materials for various high temperature tribological applications.

Keywords: MAX phases tribology; Solid lubricants; Composite lubricous materials

1. Introduction

Currently, there is a great need in modern industry for tribological systems possessing low wear rates, WRs, and low friction coefficients, μs, over a wide temperature range [1,2]. A list of potential applications includes air-foil bearings, gas turbine seals, cylinder wall/piston ring lubrication for low-heat rejection diesel engines, various furnace components, among many others [2–5].

At temperatures above ∼350 °C and, especially, in oxidizing environments, conventional liquid lubricants, and most conventional solid lubricants (e.g. graphite and MoS_2) degrade rapidly. A large number of solid materials, such as noble metals (e.g. Au, Ag, Pt), inorganic fluorides (e.g. LiF, CaF_2, BaF_2), some metal oxides (e.g. NiO, MoO_3, the Magneli phases, such as $TiO_{2-\gamma}$, etc.), have been employed as solid lubricants [3–7]. But, in general, these materials possess good lubricious properties only in a restricted temperature range and some are too brittle, especially at room temperature. Furthermore, some lubricant materials exhibit poor wear resistances and/or can cause wear of the counterparts; several are non-machinable.

To expand the applicable temperature range, composite materials containing two or more solid lubricants were developed [1–3]. Dellacorte et al. [8–10] developed a series of NiCr/Cr_2O_3 based composite coatings containing Ag (low temperature lubricant) and CaF_2/BaF_2 eutectics (high temperature lubricant) designated as PS30x. In the 26–650 °C temperature range, PS300 and PS304 coatings were reported to exhibit WRs of $\sim 10^{-4}$ mm^3/Nm and $\mu s \leq 0.4$ when tested against Ni-based SAs (Inconel 780) using a pin-on-disk tribometer [8,9]. Similar $\mu s \sim 0.4$ were reported for PS304 coatings when tested in journal air-foil bearing rigs in the 25–650 °C temperature range against Inconel 780 foil [10]. Balic and Blanchet characterized plasma-sprayed PS304 and HVOF-sprayed PS304

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coatings against Rene 41 and Inconel X-750 counterfaces in thrust-washer tests between room and 538 °C. The typical WRs were reported to be $\sim 2 \times 10^{-4}$ mm^3/Nm and μs ≤ 0.4 [11]. Recently, Heshmat et al. developed a new series of NiCr-based solid lubricates under the trade name Korolon capable of use up to 800 °C [12]. Startup/shutdown μs in 0.4–0.6 range were achieved when Korolon 1350 B coatings were tested against hard Cr, PS304 and itself in thrust-washer rigs at 650 °C. The tribological behavior of the Korolon coatings were determined to be temperature dependent with ~~minimal~~ μs < 0.1 were reported.

The tribological properties of spark-plasma-sintered $ZrO_2(Y_2O_3)$–CaF_2–Ag composites were investigated against Al_2O_3 using a ball-on-disk tribometer [2]. μs in 0.4–0.8 range were observed at temperatures between room and 800 °C.

Muratore et al. reported yttria-stabilized zirconia antifriction coatings with Ag and Mo, which were tested against Si_3N_4 counterparts in air using a ball-on-disk tribometer [3]. A $\mu \sim 0.4$ was observed for the composition YSZ–24%Ag–10%Mo in the temperature range from 25 to 700 °C.

Recently, we have shown that several ternary transition metal carbides – the so-called MAX phases, such as Ta_2AlC, Ti_2AlC and Cr_2AlC – demonstrated remarkable tribological performance when tested against Ni-based SAs at 550 °C [13] (see below). The MAX phases, having a formula unit $M_{n+1}AX_n$, where n is 1, 2 or 3, M is an early transition metal, A is an A-group (mostly IIIA and IVA) element, and X is either C and/or N, are a new class of materials which possess unusual combinations of metal and ceramic-like properties [14–16]. They are highly damage tolerant, thermal shock resistant, readily machinable, but with Vickers hardness values of 2–5 GPa, are anomalously soft for transition metal carbides and nitrides. They are also excellent electrical and thermal conductors and possess relatively low thermal expansion coefficients [16–18]. Many of these properties can be traced to the fact that basal plane dislocations are active at least down to 77 K.

In inert atmospheres, the MAX phases do not melt congruently, but decompose incongruently into a MX-based carbide or nitride and an A-rich liquid [16]. In air, some of them, most notably Ti_2AlC, demonstrate excellent oxidation resistance because of the formation of a highly protective alumina oxide layers [16,19,20].

The experimental studies that deal with the tribological behavior of the MAX phases are few and the existing ones have almost all been carried out at room temperature mostly on Ti_3SiC_2 [21–23], with a few on Ti_3AlC_2 [24].

Very recently we reported on the tribological behavior of the following MAX phases: Ti_2AlC, Cr_2AlC, Ta_2AlC, Ti_3SiC_2, Ti_2AlN, Ti_4AlN_3, Cr_2GeC, Cr_2GaC, Nb_2SnC and Ti_2SnC, tested against Ni-based SAs at 25 and 550 °C in air [13]. At room temperature, the wear rates, WRs, and μs were relatively high (WRs $\geq 10^{-4}$ mm^3/Nm and μs > 0.5). However, at 550 °C low WRs (<10^{-6} mm^3/Nm) and μs < 0.5 were observed. Their good tribological properties at 550 °C were attributed to the formation of tribo-oxides on both contact surfaces. The oxides were comprised mostly of the oxides of Ni, Cr and Fe, i.e. the SA counterpart constituents [13].

Since it is well established in the literature that Ag is a good lubricant to use at room temperature [7], in this work we attempted to improve the tribological performance of the MAX phases at room temperature by alloying them with Ag. Herein, we report on the tribological behavior of Ta_2AlC/Ag and Cr_2AlC/Ag composites tested against Ni-based SAs and Al_2O_3 in the 25–550 °C temperature range.

2. Experimental details

In order to determine the optimum Ag content in the Cr_2AlC/Ag composites, samples with different volume fractions of Ag were prepared by liquid phase sintering of cold pressed, mixed, powders of Cr_2AlC (3-ONE-2, Voorhees, NJ) and Ag (99.9%, Alfa Aesar, Ward Hill, MA) at 1200 °C for 10 min in an Ar atmosphere.

Most of the tribology experiments were carried out on Ta_2AlC and Cr_2AlC composites each with 20 vol.% Ag. These samples were hot isostatically pressed (HIPed) at 1100 °C for 20 min under ~70 MPa. The MAX phase (>92%, 3-ONE-2) and Ag (99.9%, Alfa Aesar) powders were sealed in a borosilicate glass before applying the pressure in the HIP. These samples will be henceforth referred to as TaAg11 and CrAg11, respectively.

Furthermore, a Ta_2AlC with 20 vol.% Ag composite sample, henceforth designated as TaAgR, was prepared (in collaboration with 3-ONE-2) by HIPing at 1100 °C for 20 min under 70 MPa pressure of powders sealed in steel cans.

The uniaxial compression (UC) and tensile (UT) tests were performed using a hydraulic testing machine (MTS 810, Minneapolis, MN), equipped with a controller (Microconsoler 458.20, MTS, Minneapolis, MN) and a 100 kN capacity load cell. Small 2 mm × 2 mm × 3 mm cubes were cut by a diamond wheel and used for the UC test. In all tests, a preload corresponding to a stress of about 1–2 MPa, was applied to keep the samples aligned. The dimensions of the samples for UT ~~testing~~ were 50 mm × 10 mm × 2 mm. The tensile creep studies were carried out at 550 °C under a stress of at 50 MPa, for 1 h. The strains in the latter test were measured by a capacitance extensometer (MTS, Minneapolis, MN) with a 1% strain.

The Vickers microhardness tests were carried out with a hardness tester (LECO, M-400, St. Joseph, MI) using loads between 2 and 5 N.

The friction and wear tests were performed using a high temperature tab-on-disc tribometer (CSM, Switzerland) capable of going up to 600 °C. All tests were carried out at a linear velocity of 100 cm/s and a load of 3 N, which corresponds to a stress of ≈0.08 MPa. The MAX-phase based tabs were in a form of cuboid chips (~6 mm × 6 mm × 2 mm) with flat surfaces. The counter surfaces were 9.5 mm thick cylindrical (55 mm diameter) SA discs of Inconel 718 (Inc718) (High Temp Metals, Inc., Sylmar, CA) or Al_2O_3 (CerCo LLC, OH). The base composition of the Inc718 is $Ni_{0.5}Cr_{0.25}Fe_{0.25}$, with small (<1 mol.%) ~~quantities of~~ additions like Nb, C, Mo, Si, Mn, etc.

All surfaces were polished to a 1 μm diamond finish, washed with acetone and dried, prior to testing. ~~The~~ Inc718 discs, coated with TiAlN were obtained (Honeywell, Morristown, NJ). The

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measured μs will be referred to in two ways: μ_m to refer to the mean friction coefficient over the entire sliding distance, and μ_s to refer to its steady state value during sliding.

The WRs of the MAX-phase tabs were determined by measuring their weights before and after testing. The WRs were then calculated by normalizing the volumetric wear obtained from the weight lost and the average measured densities of the composites – which were ~11.2 and ~6.1 Mg/m^3 for the Ta_2AlC and Cr_2AlC composites with 20 vol.% Ag, respectively, – by the total sliding distance and applied load. The wear of the SA discs was measured by laser profilometry [Solarius Development, Sunnyvale, CA].

For several important technological applications the tribocouples should maintain their attractive tribological properties under thermal cycling conditions. To simulate these conditions, the μs were monitored during several consecutive heating and cooling cycles from ambient to 550 °C. After each cycle, the composite tabs were removed and weighed. Surface profilometry was also carried out on the Inc718 surfaces. The pairs were then placed again in the tribometer, on the same tracks, for the next heating/cooling cycle, etc.

Two foil bearing rig tests of the MAX/Ag composites were carried out (Honeywell, Torrance, CA). Shafts for the rig tests were prepared from the CrAg11 and TaAgR samples. The shafts, 26.94 ± 0.01 mm in diameter and 37.4 ± 0.25 mm long, were machined by precision grinding (WMC Grinding Inc., Santa Fe Springs, CA). The surface roughness of the shafts was ~0.1–0.2 μm, the arithmetic average roughness (R_a). The foils used during testing of the CrAg11-SA tribocouples were chemically etched with $FeCl_3$ for 10 min prior to testing, which greatly increased their roughness, to promote ~~the~~ material~~s~~ transfer.

Both samples were tested at maximum rotation speed of ~50,000 rpm in start/stop cyclic regime to imitate the start up and shutting down conditions in a turbomachine (For the average density of the Ta/Ag composite (~11 Mg/m^3) the required tensile strengths for the shaft material to operate safely at 50 krpm was calculated to be at least 80 MPa at 550 °C (with a safety factor of 2) [25].~~)~~.

The microstructure and chemistries of the composites were characterized by field emission scanning electron microscopy (FESEM, XL-30, FEI-Philips, Hillsboro, OR) equipped with an energy dispersive spectroscope, EDS (EDAX, Mahwah, NJ).

X-ray diffraction, XRD, patterns were obtained at ambient temperatures on a powder diffractometer Siemens D500 (Bruker AXS, Madison, WI) using Cu Kα radiation, a step scan 0.02°, 1 s/step. Si powder was used as an internal standard.

3. Results

3.1. Microstructures and mechanical properties

The HIPed TaAg11, TaAgR and CrAg11 samples were >99% dense. The Cr_2AlC/Ag composite samples prepared by pressureless sintering were only ~90% dense, however. The microstructures of both composites are described elsewhere [25].

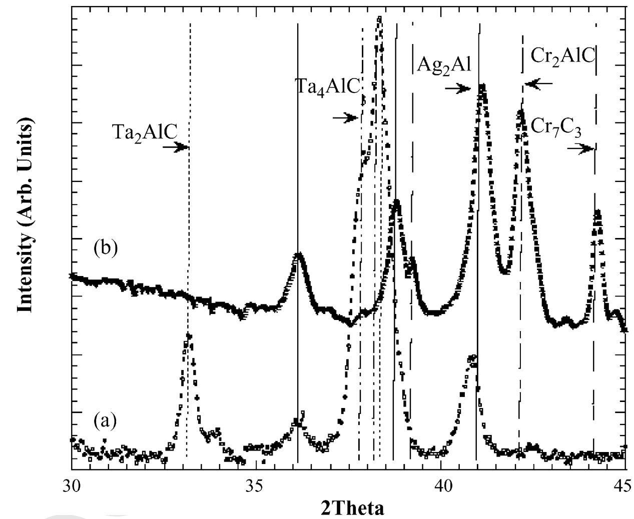

Fig. 1. XRD spectra of: (a) TaAg11 and (b) CrAg11 samples.

According to XRD ~~the~~ spectra (Fig. 1), during sintering, the MAX phases, in the presence of liquid Ag, reacted with it to form Ag_2Al, mainly in accordance to the following simplified reaction:

$$M_2AlC + Ag \rightarrow M_2Al_{1-\gamma}C + Ag_{2\pm\delta}Al$$

$$(M = Ta, Cr;\ 0 < \gamma < 1) \qquad (1)$$

EDS of the carbide grains indicated that some were Al-deficient while, according to XRD, retaining their original hexagonal crystal structure. In the case of the Ta-based composites, a slight increase in the peaks associated with Ta_4AlC_3 and Ta_2C were observed (Fig. 1); for the Cr-based those of Cr_7C_3 were increased (Fig. 1).

These composites had excellent tensile, σ_t, and compressive, σ_c, strengths in the 26–550 °C temperature range (Fig. 2). The TaAg11 composite had a $\sigma_t \sim 180$ MPa and a $\sigma_c > 1.5$ GPa at ambient temperature, and a $\sigma_t \sim 100$ MPa at 550 °C (Fig. 2a–c). When held at 50 MPa for 1 h at 550 °C, there was no discernable strain (Fig. 2d). The CrAg11 samples had a $\sigma_t \sim 150$ MPa at ambient temperature and a $\sigma_t \sim 120$ MPa at 550 °C (Fig. 2e).

The Vickers hardness of the composites was ≈5–6 GPa; that of the SA was ≈4–5 GPa. They were all readily machinable to the high tolerances ($R_a \sim 0.1$–0.2 μm).

3.2. Influence of composition on μ and WR

Fig. 3 shows the WRs and μs of the Cr_2AlC/Ag samples tested against Inconel 600 as a function of Ag volume fraction. The addition of 5 vol.% Ag reduces μ from 0.65 to 0.5. Further increases in Ag content, however, had little effect on μ. Correspondingly the addition of 5 vol.% Ag reduced the room temperature WRs by about an order of magnitude as compared to those not containing Ag. Further increases in the Ag content had little effect (Fig. 3a).

Despite the fact that the tribological properties of the MAX/Ag composites, with more than 5 vol.% Ag, depended weakly on Ag content, mechanical testing on the composites

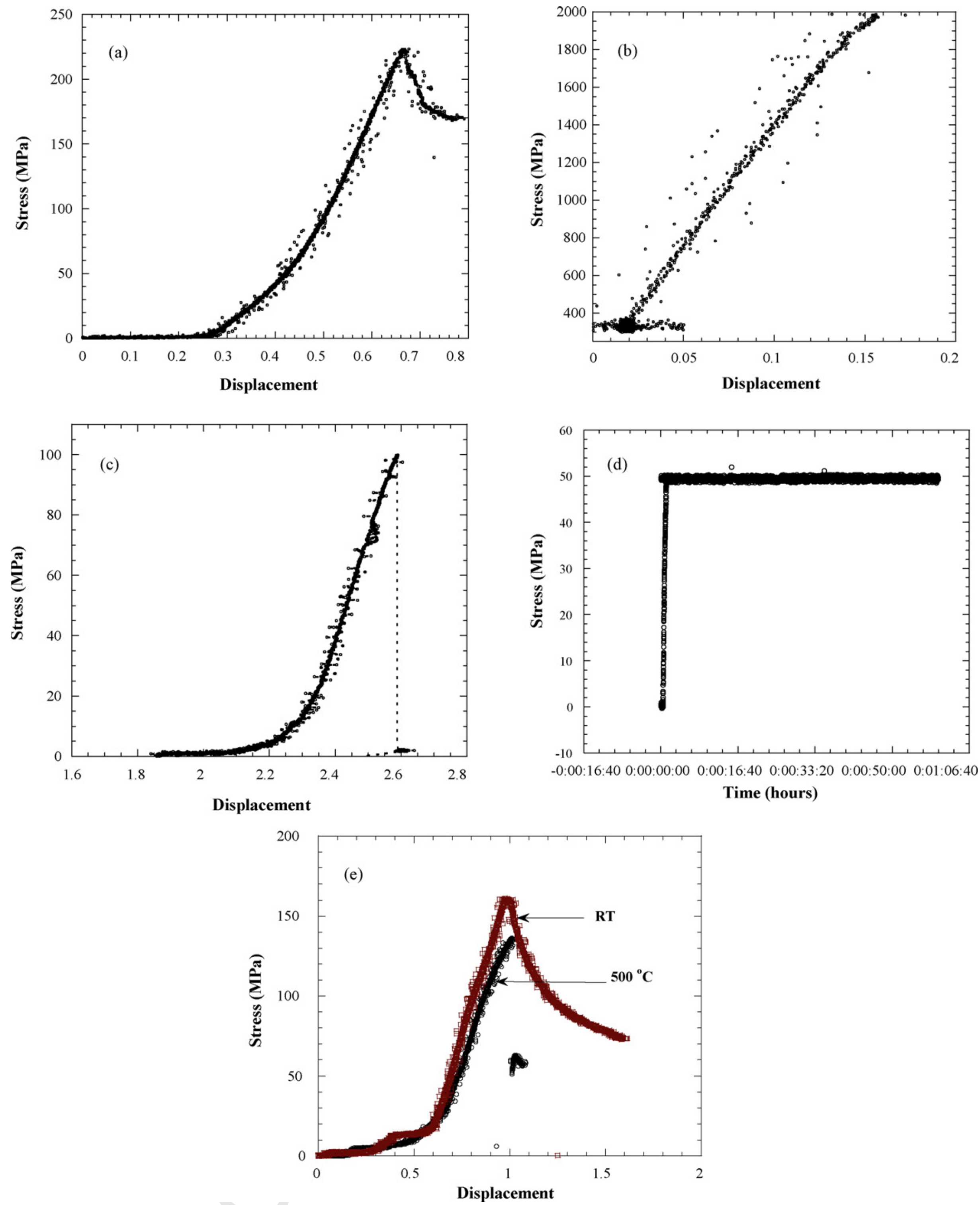

Fig. 2. (a) Tensile strength of TaAg11 at 26 °C, (b) compressive strength of TaAg11 composites at 26 °C, (c) tensile strength of TaAg11 composites at 550 °C, (d) tensile creep resistance of TaAg11 composites tested at 550 °C at 50 MPa for 1 h, and (e) tensile strength of CrAg11 at 26 °C and 550 °C.

showed that the 20 vol.% Ag samples had better mechanical properties as compared to the 10 vol.% Ag composites (Fig. 3b). Based on these results, the 20 vol.% Ag composition was chosen for further tribological testing. Higher Ag-contents were not tested because of possible degradation in the creep properties and the relatively high cost of Ag.

3.3. Effect of sliding distance on μ

3.3.1. Room temperature testing

When the TaAg11 composites were tested against Inc718, the initial μ was ~0.1, followed by a gradual increase to a μ_s of ~0.6 (Fig. 4a). Similar behavior was observed when

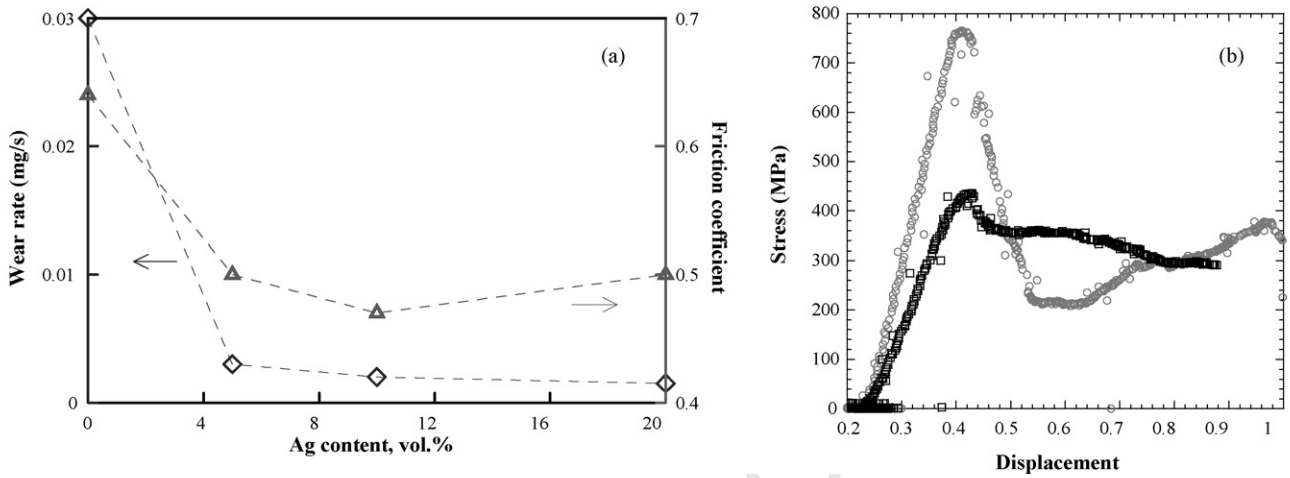

Fig. 3. (a) Wear rates (◇) and μs (△) of Cr_2AlC–Ag pressureless sintered composite samples tested against Inc600 at room temperature as a function of Ag content, and, (b) compressive strength for those samples at 26 °C with (□) 10 vol.% and (◯) 20 vol.% Ag.

this composite was tested against harder substrates, such as Al_2O_3 and the TiAlN coated samples: the μs were initially low but increased gradually to a $\mu_s \sim 0.35$ (Fig. 4b and c, respectively).

When the CrAg11 samples were tested against Inc718, μ was quite noisy, with a $\mu_s \sim 0.8$ (Fig. 4d). During testing against Al_2O_3, the initial μ was high, went through a shallow minimum, before gradually increasing to a $\mu_s \sim 0.55$ (Fig. 4e).

3.3.2. Testing at 350 °C

At 350 °C, μ_s varied between 0.2 and 0.3 for the TaAg11 sample tested against Inc718; the response was again not smooth, but was characterized by spikes during which μ increased to ~0.6 (Fig. 5a). For the Inc718/CrAg11 tribocouple, μ_s varied between 0.4 and 0.5. Here again, spikes in μ up to 0.65 were observed (Fig. 5b).

3.3.3. Testing at 550 °C

For the Inc718/TaAg11 tribocouples, the initial μ was about ~0.6 and unstable, before decreasing to a $\mu_s \sim 0.5$ (Fig. 6a). Analogous behavior was observed when this composite was tested against Al_2O_3 (Fig. 6b): the μs were initially high, before dropping to ≈0.4. Note, however, that no spikes in μ were observed in this case.

Similar behavior was also observed when the CrAg11 samples were tested against Inc718 at ~0.6, μ was initially relatively high and unstable before decreasing to a $\mu_s \sim 0.35$ (Fig. 6c).

3.3.4. Variations in μ during consecutive heating and cooling cycles

The results of the cycling tests are shown in Fig. 7. During the first heat up of the TaAg11-Inc718 tribocouple, the initial μs were low (<0.1), went through a local maximum at ≈100 °C, before increasing again to a μ_s of 0.5 at 500 °C (Fig. 7a). During the entire cooling cycle, μ varied between 0.5 and 0.6.

During subsequent heating cycles, μ again started low, before increasing to a $\mu_s \sim 0.5$ at 500 °C (Fig. 7a). During the second cooling cycle, μ was constant at ~0.5; in contradistinction to the first cooling cycle, it was much more stable (Fig. 7a). During the third heat up cycle, μ increased gradually from 0.2 to 0.4, and was fairly stable during the entire cycle. Unlike the first two cycles, μ showed no sharp transitions and the final μ was lower than during the first two heating cycles. During the third cooling cycle, μ was stable at 0.4 (Fig. 7a).

Table 1
Summary of WRs and μ of different tribocouples

Dynamic partner	Static partner	Temperature (°C)	~~Static partner,~~ WR_s (mm^3/Nm)	Dynamic partner, WR_d (mm^3/Nm)	μ_s
TaAg11	Inc718	26	2×10^{-5}	CND	0.60 ± 0.05
		350	$<10^{-6}$	CND	0.29 ± 0.04
		550	5×10^{-5}	$\sim 2 \times 10^{-4}$	0.49 ± 0.02
		26	3×10^{-5}	Deposition	0.39 ± 0.01
	Al_2O_3	350	5.5×10^{-5}	Deposition	0.45 ± 0.02
		550	6×10^{-4}	Deposition	0.42 ± 0.03
	TiAlN	26	8×10^{-6}	Deposition	0.35 ± 0.01
CrAg11	Inc718	26	1×10^{-4}	CND	0.80 ± 0.08
		350	5×10^{-5}	CND	0.41 ± 0.03
		550	7×10^{-5}	$\sim 1 \times 10^{-4}$	0.45 ± 0.03
	Al_2O_3	26	7×10^{-5}	Deposition	0.55 ± 0.01

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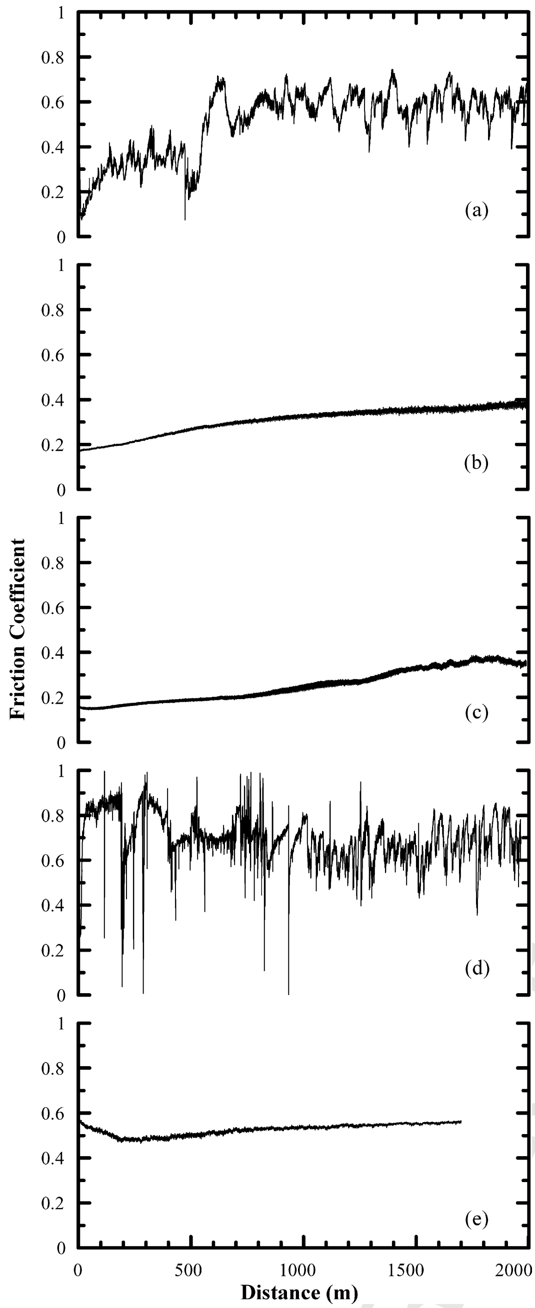

Fig. 4. Variation in μ as a function of sliding distance at ambient temperature in different tribocouples: (a) Inc718-TaAg11, (b) Al_2O_3-TaAg11, (c) TiAlN coatings-TaAg11, (d) Inc718-CrAg11, and, (e) Al_2O_3-CrAg11.

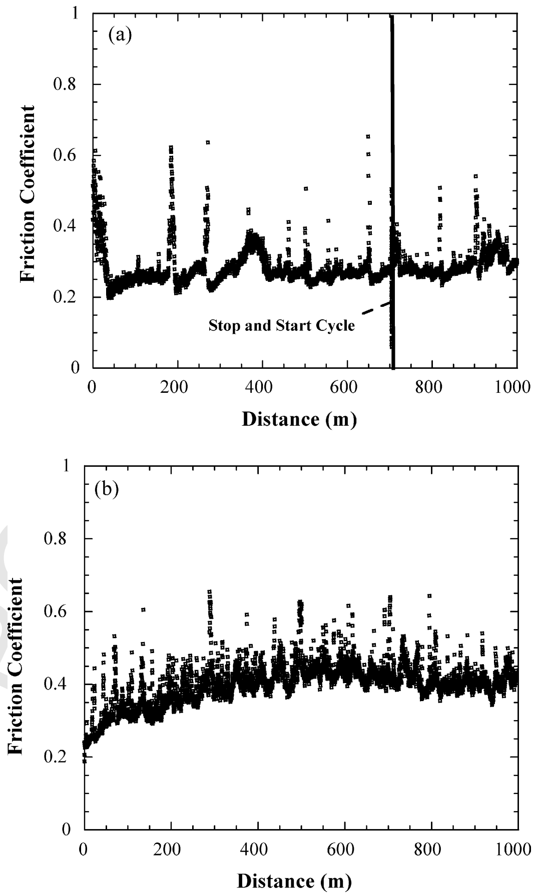

Fig. 5. Variation in μ as a function of sliding distance when Inc718 was tested at 350 °C against: (a) TaAg11, and, (b) CrAg11.

The response of the CrAg11-Inc718 tribocouple (Fig. 7b) was qualitatively similar to its Ta-counterpart. The response in this case was slightly noisier in that there were fluctuations in μ. The general trend, however, was the same: a slow but steady decrease in μ with cycling.

3.4. Wear and wear kinetics

3.4.1. Wear of tribocouples during isothermal studies

After 2 km sliding at room temperature, the WR of the TaAg11 samples was $\sim 2 \times 10^{-5}$ mm^3/Nm (Table 1). A similar WR was observed when it was tested against Al_2O_3 (Table 1). The Inc718 surface roughness, as measured by laser profilometery, is shown in Fig. 8a. No signs of large gouges were observed and the roughening observed was <0.5 μm.

At room temperature, at $\sim 1 \times 10^{-4}$ mm^3/Nm the WRs of the CrAg11 composites tested against Inc718 for 2 km were higher than those for the TaAg11 samples (Table 1).

After isothermal sliding at 350 °C for 1 km, the WR of the TaAg11 sample was quite low ($<10^{-6}$ mm^3/Nm). Here again, no gouging was observed on the Inc718 track and its WR was too low to be measured (Table 1).

After sliding for 2 km at 550 °C against Inc718, the WR of the TaAg11 composite tab ($\sim 5 \times 10^{-5}$ mm^3/Nm) was slightly higher than at room temperature (Table 1). However, the wear of the Inc718 counterpart was markedly higher than at room temperature. Valleys (gouges) and mountains (tribofilms) were observed on the tracks (Fig. 8b). To calculate the WR of the Inc718 counterpart, the wear of the tracks was modeled by assuming a uniform gouge of 2 μm over the entire track (details can be found in Ref. [13]). The WR of the Inc718 was conservatively estimated to be $\sim 2 \times 10^{-4}$ mm^3/Nm.

When the TaAg11 sample was tested vs. Al_2O_3, the wear behavior, as the temperature was increased from ambient to 550 °C, was quite different. After 2 km of sliding, the specific WR of the TaAg11 pin increased from ∼3 to 5×10^{-5} mm^3/Nm

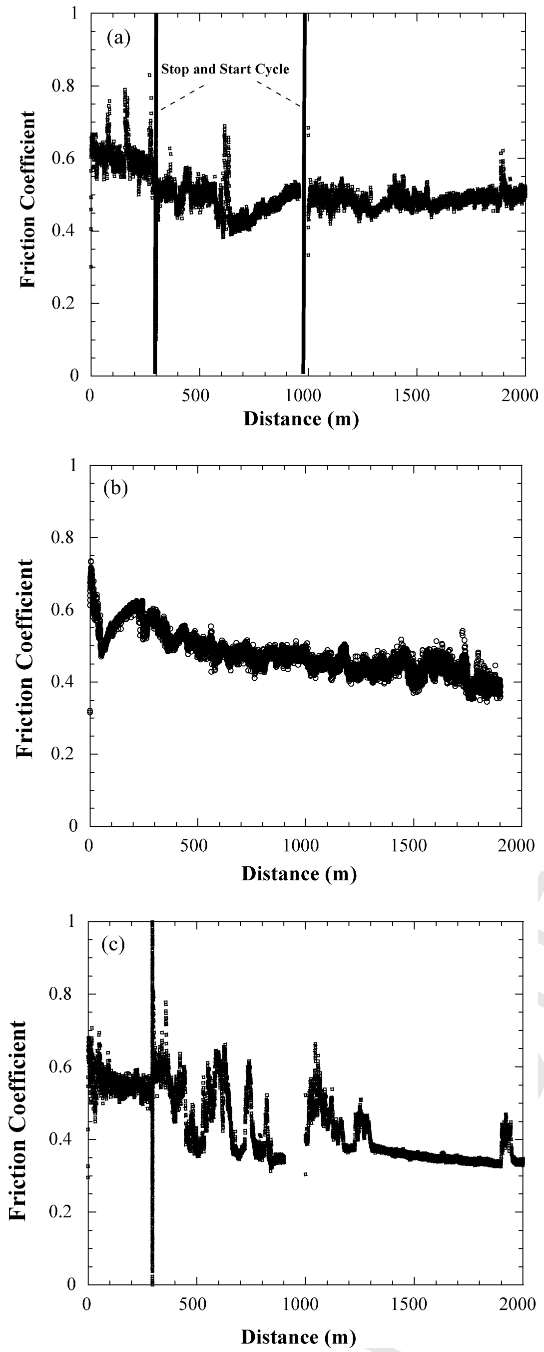

Fig. 6. Variation in μ as a function of sliding distance for different tribocouples at 550 °C: (a) TaAg11 against Inc718, (b) TaAg11 against Al_2O_3, and, (c) CrAg11 against Inc718.

in the 26–350 °C temperature range to ~6×10^{-4} mm^3/Nm at 550 °C (Table 1). For both temperatures, the corresponding wear of the Al_2O_3 disc was negligible as material was visibly transferred from the TaAg11 tab to the Al_2O_3 surface.

At ~7×10^{-5} mm^3/Nm, the WR of the CrAg11 sample tested against Inc718 at 550 °C was slightly lower than its value at room temperature. The WR of the Inc718 counterpart was estimated to be ~1×10^{-4} mm^3/Nm (Table 1).

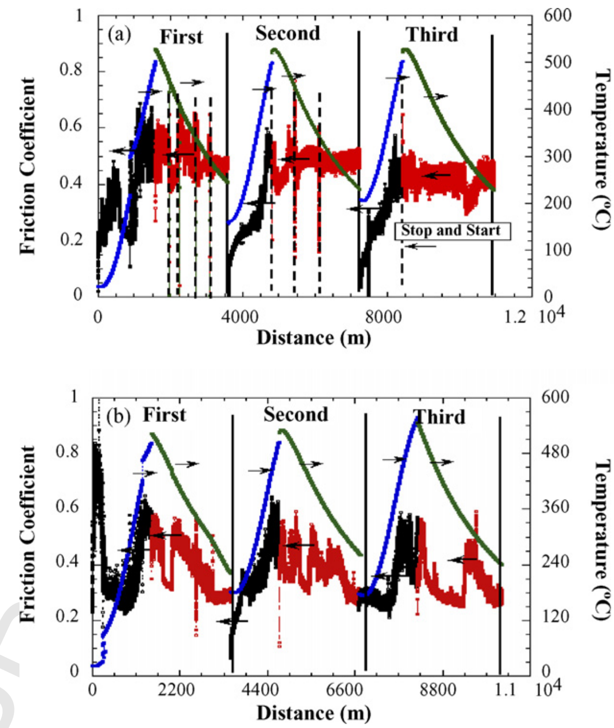

Fig. 7. Variation in μs of: (a) TaAg11-Inc718, and, (b) CrAg11-Inc718 tribocouples during heating and cooling in 26–500 °C temperature range. Black data points represent μ during heating; red, μ during cooling; blue, temperature during heating; green, temperature during cooling; black dotted line, stop and start (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.).

3.4.2. *Wear kinetics*

At room temperature, the wear of both the Ta- and Cr-containing samples was linear as a function of sliding distance (Fig. 9a). At 550 °C, the WRs of the CrAg11 and TaAg11 tabs against the Inc718 discs were quite different (Fig. 9b). For the former, the initial wear was relatively high before becoming negligible, while for the latter, the wear was linear with sliding distance (Fig. 9b).

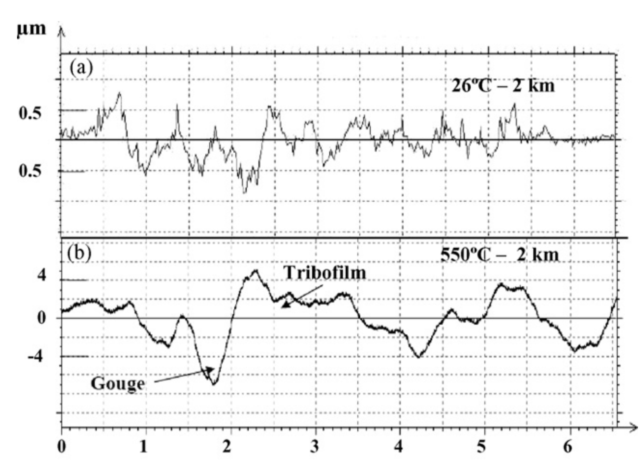

Fig. 8. Laser profilometry on Inc718 surfaces after sliding against TaAg11 at: (a) room temperature and, (b) 550 °C.

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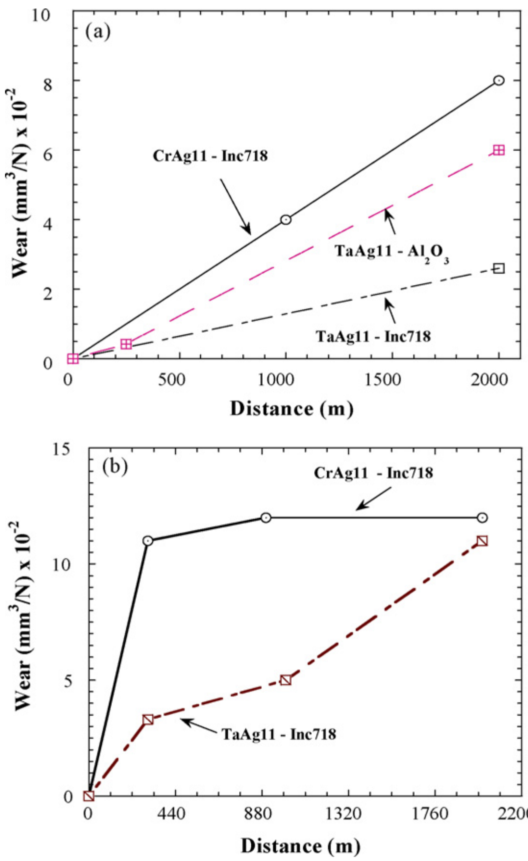

Fig. 9. Plots of wear as function of sliding distance of MAX/Ag composites against different substrates at: (a) ambient temperature, and, (b) 550 °C.

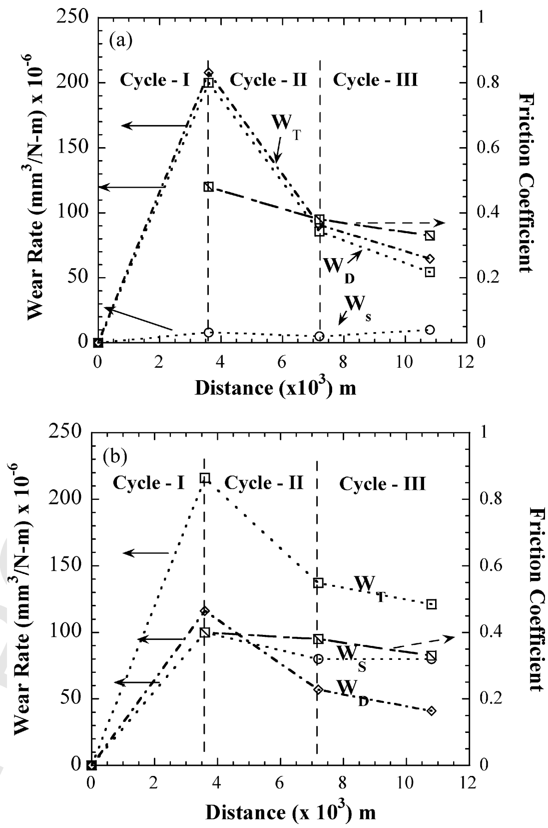

Fig. 10. WR as a function of sliding distance for: (a) TaAg11 and, (b) CrAg11 against Inc718. W_s, wear rate of static partner (Ta_2AlC/Ag or Cr_2AlC/Ag), W_D, wear rate of dynamic partner (Inc718), and $W_T = W_D + W_S$. Each data point represents 1 heating and cooling cycle.

3.4.3. Wear of tribocouples during thermal cycling

During the heat/cool cycling of the TaAg11-Inc718 tribocouple with a sliding distance of 3 km/cycle, the WR of the TaAg11 sample varied between 8 and 10×10^{-6} mm^3/Nm. The corresponding WR of the Inc718 counterpart was $\sim 2 \times 10^{-4}$ mm^3/Nm after the first heat/cool cycle; after 11 km it had decreased to $\sim 6 \times 10^{-5}$ mm^3/Nm.

The total (both counterparts) cumulative WRs of the TaAg11-Inc718 tribocouples under thermal cycling conditions are shown in Fig. 10a. The total cumulative WR after the first heat/cool cycle was 2×10^{-4} mm^3/Nm, which value then decreased to 7×10^{-5} mm^3/Nm after 11 km of total sliding, i.e. after the 3rd heat/cool cycle (Fig. 10a).

The corresponding results for the CrAg11/Inc718 tribocouple are shown in Fig. 11b. The total cumulative WR after the first heat/cool cycle was 2×10^{-4} mm^3/Nm and decreased slightly to 1×10^{-4} mm^3/Nm at the end of third heat/cool cycles (Fig. 10b).

3.5. Foil bearing rig tests

The TaAgR and CrAg11 samples were tested in an air-foil bearing rig setup against Ni-based SA foils. In both cases, the initial stop/start cycles were carried out at 350 °C. For the TaAgR sample, the tribocouple was tested at that temperature for 5050 stop–start cycles; the temperature was then increased to 550 °C

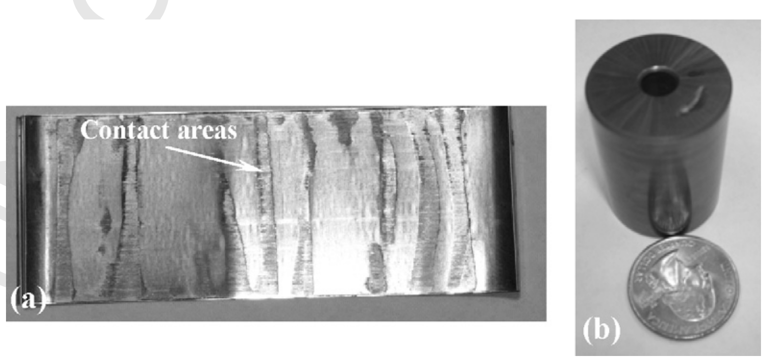

Fig. 11. Pictures of: (a) Ni-based SA top foil, and, (b) TaAgR shaft after testing in the air-foil bearing rig.

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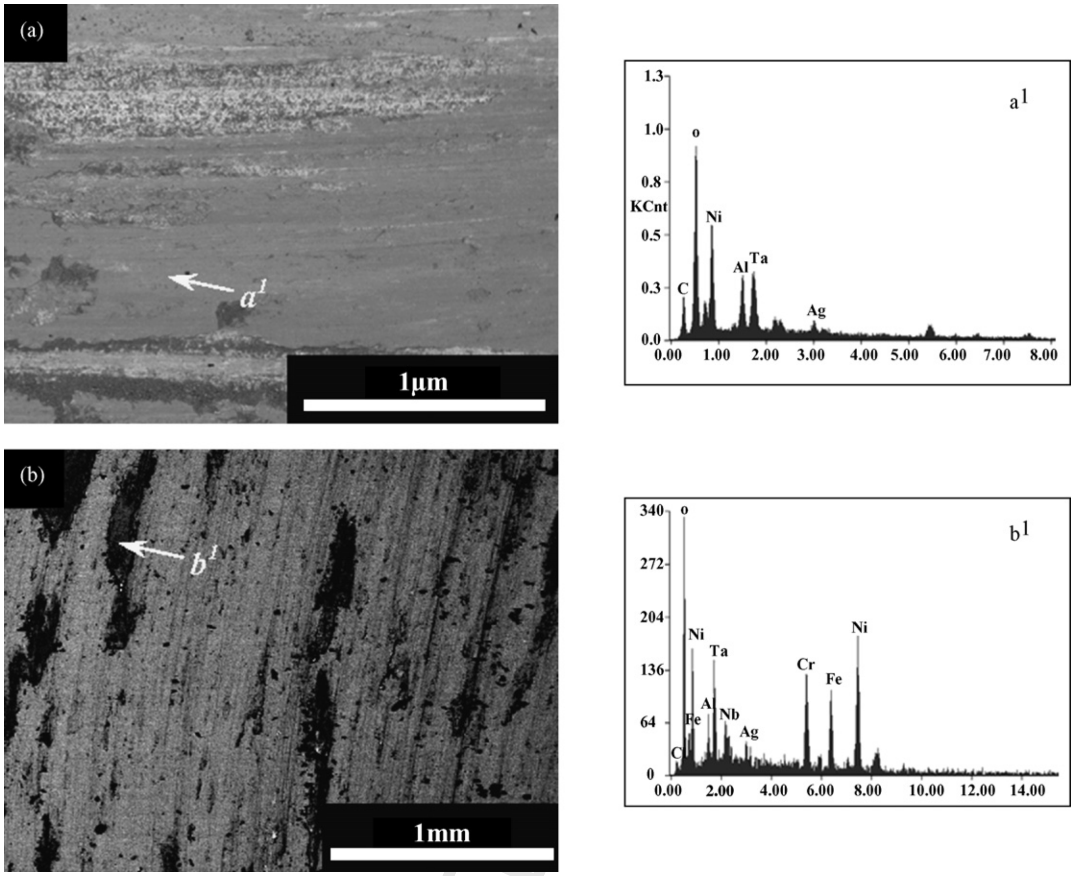

Fig. 12. BS SEM micrographs of the worn surfaces of TaAg11 samples after 2 km sliding against Inc718 at 550 °C and typical EDS in the selected locations: (a) TaAg11 surface and (b) Inc718 counterpart.

for 2370 cycles, and finally the setup was cooled to room temperature and tested for 2590 cycles. After the completion of the ∼10,000 cycles, dark areas were observed on the SA foils contact surfaces (Fig. 11a).

Before testing, the surface roughness of the SA foil, R_a, was 0.2 µm; after 10,000 cycles it increased to 0.4 µm in the contact areas. Before the rig test, the MAX/Ag surface was machined to a finish of R_a of 0.1 µm; after testing R_a increased to 0.2 µm, but the surface was still optically reflective (Fig. 11b).

The CrAg11/SA tribocouple in the foil bearing system was initially tested at 350 °C for 1000 cycles, then at 550 °C and lastly at 25 °C for 1000 cycles each, for a total of 3000 cycles. Similar to the TaAgR samples, the CrAg11 shaft were micro-smooth after testing. The roughness of the SA foil was ∼1 µm R_a; a value that was relatively high because, as noted above, the foils were intentionally roughened by etching.

4. Discussion

It was reported recently that a number of the MAX phases, i.e. Ta_2AlC, Ti_2AlC, etc. were found to be good self-lubricating tribological materials when they were tested against Ni-based SAs at elevated temperatures [13]. However, they had high WRs at room temperature caused by a third-body abrasion. Addition of Ag prevented the wear particle formation and, thus, greatly reduced the WRs of the MAX/Ag composite materials at room temperature as compared to the pure MAX phases. Note that Ag reacted with MAX phases and, therefore, the main Ag-containing metal phase was $Ag_{2\pm\delta}Al$. Simultaneously, the addition of Ag slightly increases the WRs of both the MAX/Ag composite and Inc718 counterparts at 550 °C, which, nevertheless, remain low for both composites. For example, the WRs of pure Ta_2AlC and Cr_2AlC tested against Inc718 were $\sim 10^{-6}$ mm^3/Nm [13], while the WRs of the MAX/Ag composites tested under the identical conditions were determined to be $\sim 10^{-5}$ mm^3/Nm (Table 1). The WRs of the Inc718 counterparts after the testing against the pure MAX phases and MAX/Ag composites were $\sim 10^{-5}$ and $\sim 10^{-4}$ mm^3/Nm, respectively. Note that the lowest WRs in both the MAX/Ag-Inc718 tribocouples were observed at the intermediate temperature of 350 °C.

A similar trend was observed when the results of the $Ta_2AlC/Ag–Al_2O_3$ tribocouples were compared to those not containing Ag. Again while the addition of Ag significantly reduced the WRs of the composite at room temperature, at 550 °C pure Ta_2AlC demonstrated lower WRs against Al_2O_3 than the Ta_2AlC/Ag composite. No visible wear of the Al_2O_3 counterparts was detected at any temperature.

Almost in all cases, formation of transfer films on the contact surfaces was observed. When Ta_2AlC/Ag or Cr_2AlC/Ag composites were tested against Inc718, clearly visible tribofilms were formed at elevated temperatures and during thermocycling. The tribofilms formed during testing at 550 °C were discontin-

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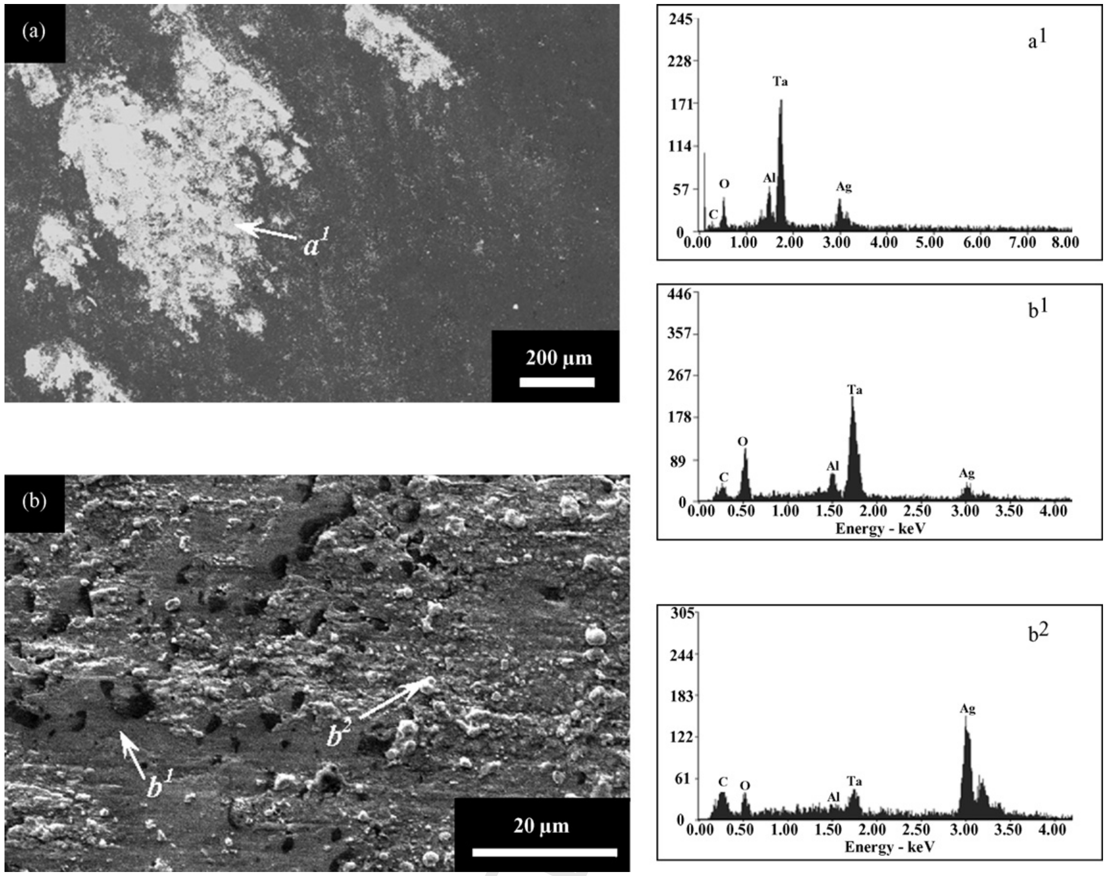

Fig. 13. SEM micrographs of the surfaces of Al_2O_3 counterparts after 2 km sliding against TaAg11 at: (a) room temperature (BS) and, (b) at 550 °C (SE)~~, and typical EDS in the selected locations.~~

uous (Fig. 12a and b), while the MAX contact surfaces after the thermocycling testing were almost entirely covered with tribofilms. According to the EDS analysis of worn surfaces, all the tribofilms formed consisted of oxidized components of both Inc718 and composite counterparts in slightly different proportions (Fig. 12a and b). At room temperature, there was no visible tribofilms on the contact surfaces, but EDS analysis revealed oxidized layers on the MAX counterpart surfaces.

The tribofilms formed during sliding against Al_2O_3 were clearly visible at all temperatures, even at ambient temperature (Fig. 13a and b), at the same time the tribofilms formed at elevated temperatures were almost continuous (Fig. 13b). At any temperatures, according to EDS analysis, the tribofilms were comprised of oxidized components of MAX/Ag composites (Fig. 13, a1- and b1-areas). At room temperature, the tribofilms were compositionally quite uniform, but at 550 °C partial melting and separation of the metallic Ag-rich phase were observed (Fig. 13b, b2-area).

As was shown for the pure MAX phases [13], here also the oxidized tribofilms formed by friction on the contact surfaces apparently are lubricous. The fact that the WRs, and to some degree, the μs, both decrease during thermal cycling (Fig. 7) must be attributed to compositional changes and self-adaptation of the tribofilms during sliding. In contradistinction, the high WRs against the Al_2O_3 counterparts at 550 °C presumably results from the tribofilm's degradation. A detailed microstructural and microchemical analysis of the tribofilms formed herein will be reported elsewhere [25,26].

For both the Ta/Ag and Cr/Ag samples tested against SAs, the addition of Ag resulted in large fluctuations in the μs (Figs. 4–6) that were not present when no Ag was present. In contradistinction, the μs were stable, i.e. no slip–stick was observed, when tested against Al_2O_3 over the entire temperature regime tested (Figs. 4b and 6b). These results clearly indicate that the transfer films that form on alumina have quite different properties than the ones that form on the SA. This conclusion was confirmed by the detailed microstructural and microchemical analysis reported elsewhere [25,26].

Summarizing the tribological properties of the new MAX/Ag composites developed herein one may conclude that these materials demonstrated relatively low and stable value of WRs and μs in the temperature range from ambient to 550 °C as well as during thermocycling. In addition, they possess decent mechanical properties and are readily machinable, which made possible to test them ~~in the journal~~ air-foil bearing rig in configuration where MAX/Ag composites were machined as bushings on ~~the~~ shafts. Both ~~the~~ samples passed extended tests at ∼50 krpm and 500 °C without significant creep. ~~T~~The surface roughness of the MAX/Ag bushings after the tests ~~at ∼0.2 μm~~ was relatively low. The roughness of the foil counterparts were higher, but intermediate measurements revealed that there was some roughness reduction, i.e. self-polishing of worn areas during testing at the

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different temperatures. Similar to the tribometer testing results, visible tribofilm formation in the contact areas was also observed on the foil surfaces.

5. Conclusions

The tribological performances of Ta_2AlC and Cr_2AlC composites with 20 vol.% Ag were investigated in the room temperature up to 550 °C temperature range, during sliding against Ni-based SA and Al_2O_3. The MAX/Ag composites demonstrate much improvement in wear resistance at low temperatures as compared to the pure MAX phases. Simultaneously, their performance at elevated temperatures was slightly worse. More importantly, the tribological performance of the MAX/Ag composites appeared to improve with both sliding distance and thermal cycling. This improvement can be presumably attributed to the oxidized tribofilms which were formed during friction. Their formation was observed ~~as~~ in tribometer experiments as well as ~~in the~~ air-foil bearing testing.

The tribological performance of the composites described here, along with their decent mechanical properties and ease of machining to very high tolerances, allows to conclude that these materials have great potential for various high temperature tribological applications. They are particularly suitable for ~~that kind of~~ tribological applications where the use of coatings is undesirable.

Acknowledgement

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