

# Tribological behavior and wear surface analysis of metal-matrix composites

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This paper describes the results of dry sliding wear tests and wear surface analysis of boron-fibre-reinforced aluminium composites. The wear tests were conducted for normal and parallel orientations, rubbing against a rotating steel disc at different loads at speeds of 0.6 and 1.6 m s<sup>-1</sup>. In addition, the wear surface analysis of the aluminium-based composites was investigated by X-ray photoelectron spectroscopy (XPS). The metal-matrix composites showed excellent wear resistance compared with the unreinforced matrix. Fibre orientation had a small effect on wear rate: at low speed the normal orientation gave the lowest wear rate, with the performance of the parallel orientation being slightly worse, but this trend reversed at high speed. The XPS analysis revealed that there was no significant variation in the amount of oxide over the surface under the test conditions used. At low speed in the normal orientation boron oxide and iron boride were formed, while at high speed the proportions of these components were slightly different and showed alloyed material with more iron boride and aluminium but less boron in total. © 1999 Kluwer Academic Publishers

## 1. Introduction

Metal-matrix composite materials (MMCs) have been studied for two decades and their properties are becoming increasingly well known. The unidirectional fibre-reinforced MMCs have shown improved mechanical properties at room and elevated temperature [1]. In previous studies, the dry wear behaviour of the boron-reinforced aluminium alloy composites was compared with those of 2014 aluminium alloy matrix. Worn surfaces were examined by scanning electron microscopy (SEM) to determine the predominant wear mechanism. It was shown that the adhesion or delamination theories of wear could explain the behavior of the matrix, in which fibres were worn down by chipping of protruding fibre ends when the wear surface was normal to the fibres axes [2–4].

In a more recent paper, the effect of fibre orientation on the dry sliding wear of continuous boron-fibre-reinforced aluminium alloy composites was studied [5]. The MMCs samples were tested in normal, parallel and anti-parallel orientations sliding against a steel counter-disc at a fixed speed of 1.0 m s<sup>-1</sup> under different loads. The results showed that for the matrix alloy and the MMCs the average wear rate increased linearly with load. The wear of the MMCs was insensitive to the fibre content, but for composites with a fibre content at or above the minimum of 16 vol % used for that work caused a large reduction in the wear rate, and thereafter no significant variation occurred. Fibre orientation had a minor effect on wear rate: the N orientation gave the lowest wear rate with the AP orientation slightly

higher and the P orientation significantly higher. It was concluded from these results and a metallographic examination that the mechanism of wear of MMCs was essentially oxidative wear of the matrix. The hard fibres modified this to slightly different degrees depending on their orientation relative to the wear surface and sliding direction.

In this further study, the wear tests were carried out in the N and P orientations at speeds of 0.6 m s<sup>-1</sup> and 1.6 m s<sup>-1</sup>. The X-ray photoelectron spectroscopy (XPS) technique was used to identify the elements present in the worn surfaces of the composite pin-samples produced under similar test conditions after the wear tests.

## 2. Experimental procedure

### 2.1. Materials

The MMCs were made by a liquid metal infiltration process in which liquid aluminium alloy was injected into a heated die containing pre-placed parallel layers of unidirectional boron fibre tape. These fibres had an overall diameter of 140 µm which included a tungsten core 15 µm in diameter and a surface coating of silicon carbide 2.5 µm thick. The plate-shaped sample produced by infiltration contained 0, 16, 21, 23, 27 and 32% by volume of boron fibres, identified by the names Unreinforced (A1), B16, B21, B23, B27 and B 32, respectively.

Portions of these composite plates were removed and machined to form the cylindrical ends of wear pins, 6.33 mm in diameter and about 4 mm thick. This was

too short to fit into a standard pin-on-disc machine. To form a pin of the necessary length the cylinders were bonded to a 50 mm long steel extension pin of the same diameter, using an epoxy adhesive, with a brass sleeve fitted over the joint for extra strength. The pin was then maintained in a brass holder in the wear machine so that it was held firmly perpendicular to the flat surface of the rotating counter-disc. This was followed by a polishing procedure using a grinding paper up to 800 grade. The disc surfaces were flatly ground to give a surface finish of approximately  $0.15 \mu\text{m}$  (CLA). The pin and disc were cleaned ultrasonically and weighed in a microbalance before each test, and then inserted into the machine. Two orientations of samples were chosen so that normal surfaces of the plates identified by the suffix "N" and parallel surfaces identified by the suffix "P" were in contact with the En 31 steel disc during wear testing. Details of the manufacture and test procedures are given elsewhere [1].

The tests were carried out at sliding speeds of 0.6 and  $1.6 \text{ m s}^{-1}$  for a period of 2 h under various loads at room temperature. The end of each pin was then cut off and the wear surface was examined by the XPS technique. After the test, the specimens were cleaned and dried, and the wear surfaces were gold-coated before examination.

## 2.2. X-ray photoelectron spectroscopy (XPS)

XPS was used to identify the elements present in both normal- and parallel-oriented worn surfaces of the composites after wear tests. In this technique, the samples were irradiated with monochromatic x-rays which produce photoelectron emission from atoms very near to the surface. Measurement of the photoelectron energies allows the identification of elements in the near-surface region and this provided information on the chemical compounds present.

## 3. Results and discussion

### 3.1. Wear

The total mass loss over the whole period of the test was measured, and for each oriented composite specimen the volumetric wear rate per unit sliding distance was calculated from the known density measurements. The average wear results of the tests carried out on the N and P orientation composites and their matrix at different loads are presented in Figs 1a and b and 2a and b for sliding speeds of 0.6 and  $1.6 \text{ m s}^{-1}$ , respectively. Each wear volume result represents the mean of at least four tests.

According to the results shown in Fig. 1 for the MMCs tested at a speed of  $0.6 \text{ m s}^{-1}$ , at all loads the composites reinforced with unidirectional boron fibres showed better wear resistance than the unreinforced alloy. For example, the average volumetric wear rate for the unreinforced matrix was calculated to be about  $1.415$  and  $4.525 \text{ mm}^3 \text{ km}^{-1}$  under 22 and 42 N load respectively. For the composites, however, it was approximately  $0.64$  and  $1.183 \text{ mm}^3 \text{ km}^{-1}$  for the N orientation, while it was about  $0.733$  and  $1.391 \text{ mm}^3 \text{ km}^{-1}$  for the

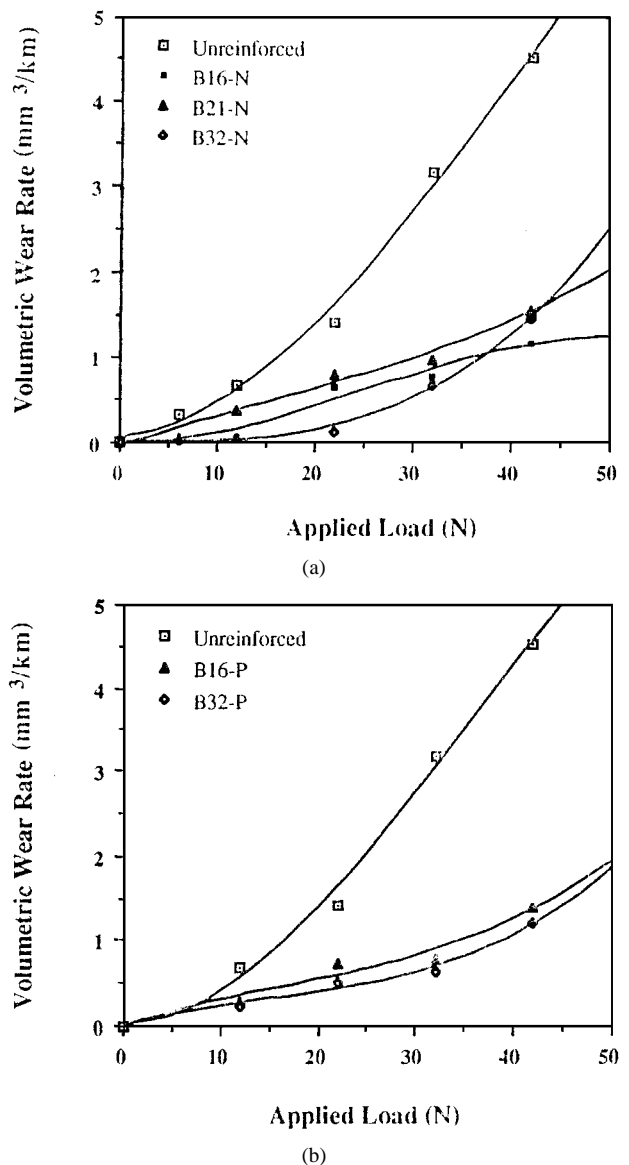
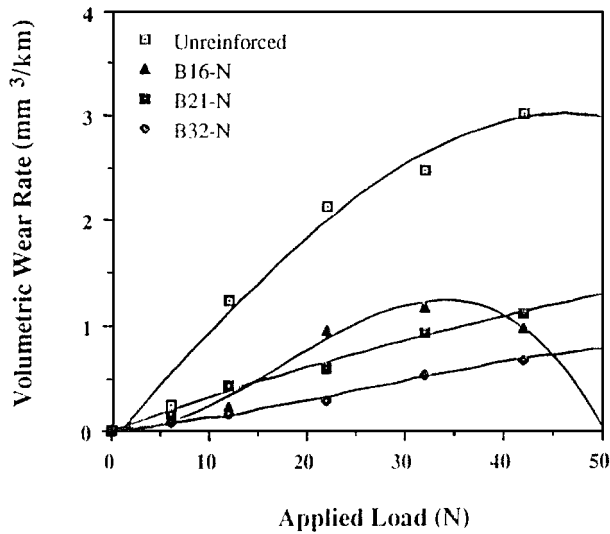
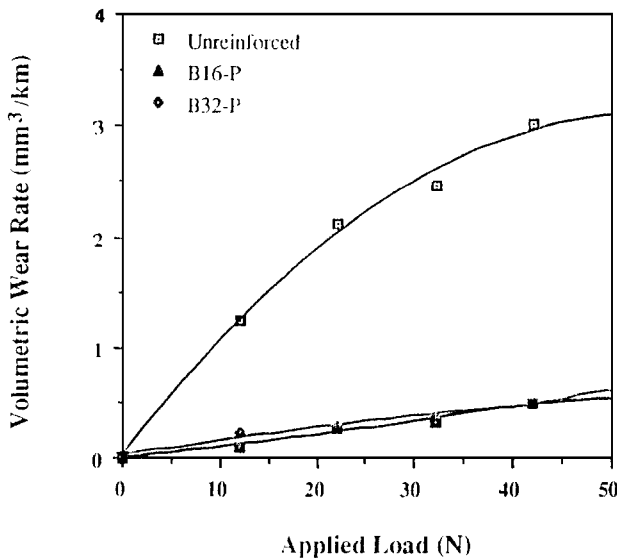


Figure 1 Average volumetric wear rate against applied load for boron fibre-reinforced aluminium composite materials at a speed of  $0.6 \text{ m s}^{-1}$  tested (a) in the N orientation, (b) in the P orientation.

P orientation under the same test conditions. Similar reductions were also obtained for the other load conditions because of continuous hard fibre reinforcement. As can be seen from Fig. 1, the volumetric wear rate increased as the normal load increased in both cases of the composites under similar conditions. But a considerable difference was found between the lowest volume fraction of fibre composite and the unreinforced matrix alloy. The increasing non-linear behaviour of the composites and their difference with the matrix was probably caused by the fibres in the matrix breaking on exposure at the rubbing surface due to the change in the surface characteristics of the specimens [2]. In addition, it was observed that the fibre orientation slightly influences the wear rate of the composite, which is lower when the fibres are in the N orientation rather than in the P orientation. In a previous study it was shown that for these composites the exposed ends of the boron fibres had been chipped at the edges during the wear [5]. For the P orientation, the wear rate again increased with increasing normal load under similar conditions.



(a)



(b)

Figure 2 Average volumetric wear rate for boron fibre-reinforced aluminium composite materials at a speed of  $1.6 \text{ m s}^{-1}$  tested (a) in the N orientation, (b) in the P orientation.

However, the counter material in this case bore against long fibres lying in the surface, with the sliding direction aligned axially along the smooth surfaces of these fibres. Some of these fibres were removed entirely from the surface by sliding out of their grooves, especially at low speed.

Fig. 2 shows the average volumetric wear rate for the MMCs tested in the N and P orientations, respectively, at a speed of  $1.6 \text{ m s}^{-1}$ . There was an increase in wear rate with load for the matrix alloy below 44 N and a decrease thereafter. It appeared that at high pressures the intermetallic  $\text{CuAl}_2$  particles may get smeared over the surface and so produce a change in the trend of wear rate with load [1]. For the composites, linear increases in wear rates could be seen with load, except for the 16% fibre-reinforced composite, and a large scatter in volumetric wear rates was observed between the various volume fraction of fibres. The 16% fibre composite showed similar characteristics to the unreinforced matrix, but a large difference was still evident between

the matrix and this composite with low volume fraction of fibre. This might be due to an uneven distribution of fibres in the composite sample. However, the 21% and 32% fibre composites showed similar trends of monotonic increases with increasing load up to 32 N, but the 21% fibre composite generated higher wear than the 32% composite. This is probably because of the small number of about 5% differently oriented fibres in this bidirectional material [5].

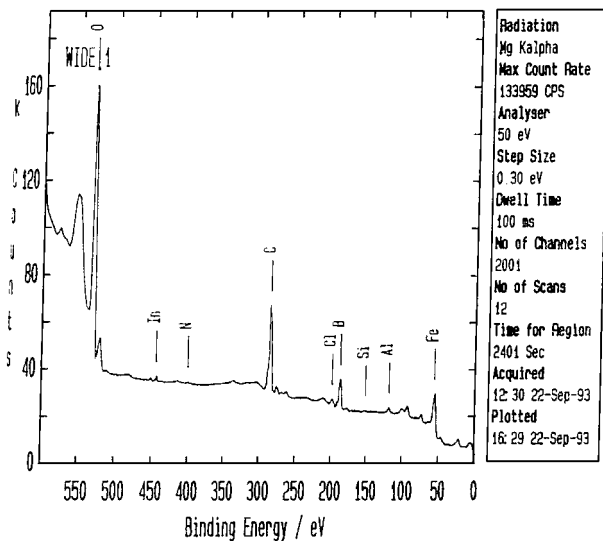
For the P orientation, it is clear that no fibre volume fraction effect on the dry wear of the composites could be observed. The wear resistance of the composites was about six times higher than that of the base alloy and the wear rate of the P orientation was found to be lower than that of the N orientation. This is probably because the fibres were loaded as cantilevers under the action of the frictional force in the N sliding direction. The friction force produced larger deflections and stresses in the fibres. Increasing sliding speed increased the lateral impact cycle and thereby resulted in chattering. The chattering also accelerated debonding and fracturing of the boron fibre [1, 5]. For the P orientation, however, the deflections and stresses were not so large in comparison to the N orientation, as the fibre remain supported by the matrix. Moreover, fibre breakage took place in the surface and subsurface, producing many short segments of fibres at this speed. Thus it was difficult to remove whole fibres from their place resulting in longer times to achieve this, in contrast to the low speed due to pulling-out of fibre at that condition. In general, the data show that the continuous boron fibre was very effective in reducing the wear rate of the matrix alloy. The wear rate increased with increasing load. The effect of fibre orientation on the dry wear was small for the composites, but very large for the unreinforced matrix alloy.

### 3.2. X-ray photoelectron spectroscopy on the worn surface

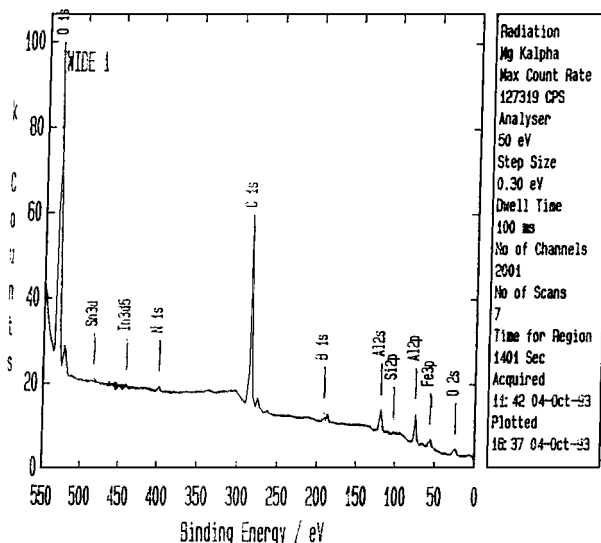
The worn surfaces of the MMCs were analyzed by XPS to determine the chemical nature of the surface compositions. Tests were conducted under similar conditions only for the N and P orientation of the 32% fibre composite. Typical XPS spectra are shown in Figs 3–5 and the chemical composition of the worn surface is listed in Table I.

TABLE I Chemical composition of a worn surface formed during dry sliding wear

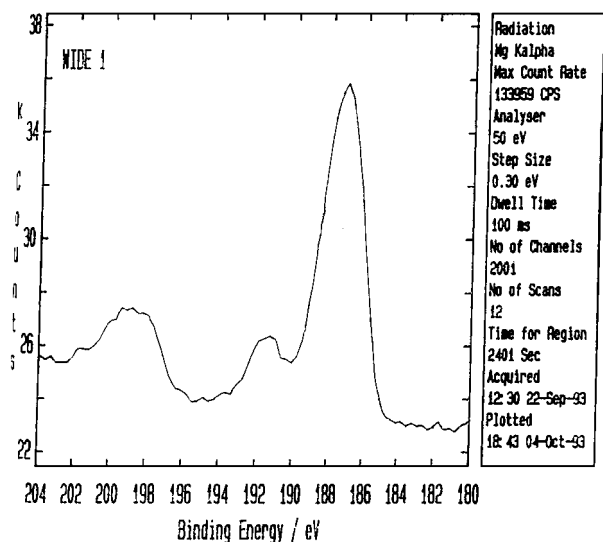
Level of peak number	Element identified	N orientation at $0.6 \text{ m s}^{-1}$ (%)	N orientation at $1.6 \text{ m s}^{-1}$ (%)	P orientation at $1.6 \text{ m s}^{-1}$ (%)
1	Al 2s	3.44	12.45	14.06
2	Si 2s	1.17	—	—
3	B 1s	21.46	9.38	12.12
4	Cl 2p	0.96	—	—
5	C 1s	29.06	43.00	38.05
6	N 1s	0.54	0.74	0.68
7	O 1s	33.89	32.12	34.09
8	Fe 2p <sub>3</sub>	9.50	2.20	0.91
9	Sn 3d	—	0.06	0.04
10	In 3d <sub>5</sub>	—	0.05	0.04



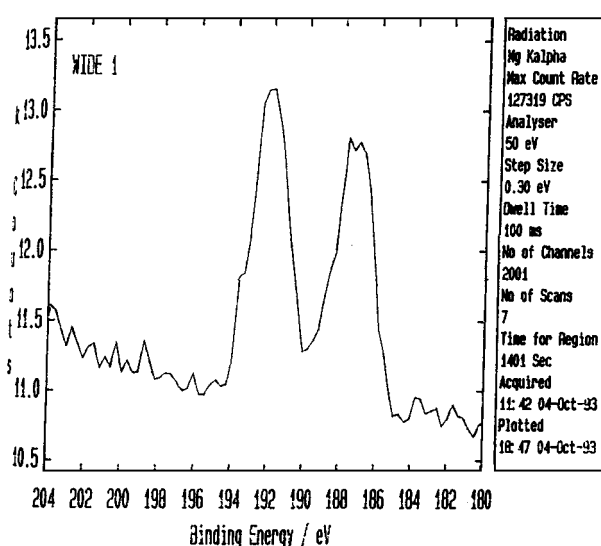
(a)



(a)



(b)



(b)

Figure 3 (a) XPS spectrum of a worn surface of a composite pin sample tested at a speed of  $0.6 \text{ m s}^{-1}$  and a load of  $22 \text{ N}$  in the N orientation, (b) showing the expanded area of (a).

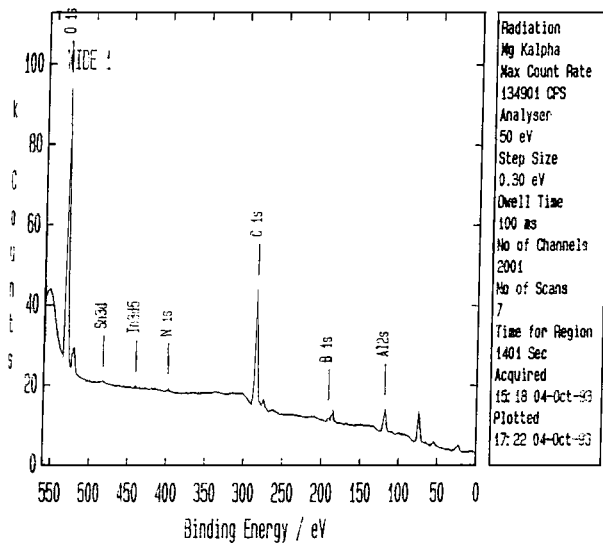
Figure 4 (a) XPS spectrum of a worn surface of a composite pin tested at a speed of  $1.6 \text{ m s}^{-1}$  and a load of  $12 \text{ N}$  in the N orientation, (b) showing the expanded area of (a).

Fig. 3a shows the XPS plot recorded for a worn pin tested in the N orientation, at a speed of  $0.6 \text{ m s}^{-1}$  under  $22 \text{ N}$  load. This figure shows that O, C, B, Al, Fe, Si and N were present. The oxygen peak was more intense than the others; carbon, iron and boron gave small peaks but the others were very small. Of these elements, the oxygen and nitrogen were from the atmosphere, and the presence of carbon was due to unavoidable atmospheric contamination or to residues of sample cleaning. Iron picked up from the counter-face, boron from the reinforcement and aluminium from the matrix were detected. From this figure, it can be seen that there was no significant variation in the amount of oxide over the surface under these sliding conditions and orientations.

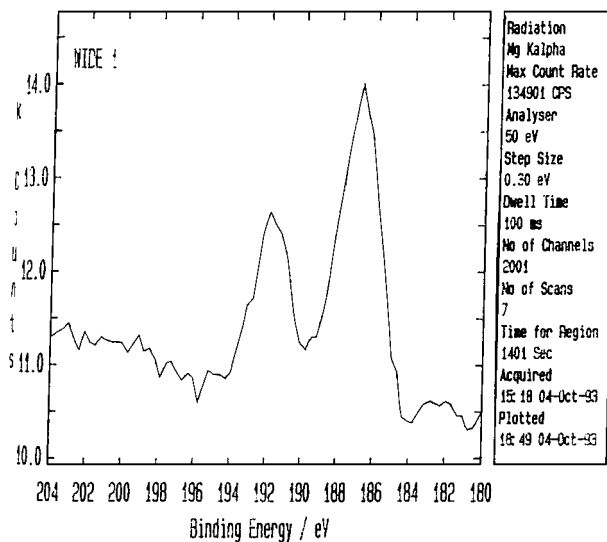
Fig. 4a shows similar spectral information from the N orientation sample but tested at a speed of  $1.6 \text{ m s}^{-1}$  under  $12 \text{ N}$  load. This spectrum were chosen from a selection of the experiments to illustrate the dependency on such factors as load and sliding speed. Similar elements were detected on the worn surface of the pin,

but the carbon peak was relatively high in the case of this orientation. Fig. 5a shows a spectrum of a similar sample but tested in the P orientation at a speed of  $1.6 \text{ m s}^{-1}$  under the same load. The spectra of all the elements were generally the same as for the N orientation, except that the boron and aluminium peaks were more pronounced and oxygen and carbon was predominant in this sample.

The numerical results obtained by XPS were recorded and showed similar trends to those shown in the above figures. However, a more in-depth look was taken of these spectra with respect to the effect of fibre orientation. The expanded parts of the graphs are shown in Figs 3b, 4b and 5b respectively. Fig. 3b shows a greatly expanded part of the recorded spectrum of Fig. 3a. According to Fig. 3b, basically two boron components containing boron oxide ( $\text{B}_2\text{O}_3$ ) and iron boride ( $\text{FeB}_2$ ) were formed, corresponding to binding energy peaks of about  $36$  and  $26.2$  amplitude (in k units of  $2 \text{ kc}$  and  $13 \text{ kc}$ ), respectively. Apart from oxygen, the main elements present on the worn surface of the



(a)



(b)

Figure 5 (a) XPS spectrum of a worn surface of a composite pin run at a speed of  $1.6 \text{ m s}^{-1}$  and a load of 12 N in the P orientation, (b) showing the expanded area of (a).

sample were Fe, B, Al and C. As atomic percentages, the amounts of the elements in this XPS plot were as follows: Fe, 9.49%; B, 21.56%; Al, 3.4%; and C, 29%, as shown in Table I. The expanded area of the N oriented sample in Fig. 4a is shown in Fig. 4b. Tested at a speed of  $1.6 \text{ m s}^{-1}$  and under 12 N load, the components of boron oxide and iron boride appeared again, but the amounts of these components were quite different, i.e.  $\text{B}_2\text{O}_3$  had about the same amplitude of 2 kc but  $\text{FeB}_2$  had decreased by a factor of 6. Also, the total amount of boron decreased from 21.46% to 9.38% and that of Fe decreased from 9.5% to 2.2%. However, Al and C increased significantly from 3.4% to 12.45% and from 29% to 43%, respectively. This was the feature of the high speed and low load test condition. Fig. 5b shows the expanded area of the P oriented sample in Fig. 5a, tested at a sliding speed of  $1.6 \text{ m s}^{-1}$  and 12 N load. The same two components were also observed here. Boron oxide remained at 2 kc peak amplitude as in the other samples, whereas iron boride had increased slightly to a 3.4 kc amplitude.

It is worth comparing the results observed here with those of similar materials tested, especially with respect to the effect of fibre orientation on graphite fibre-reinforced metal-matrix composites. For example, Eliezer *et al.* [6] studied the dry sliding wear of graphite-aluminium composites, and the lowest wear rate was obtained when the fibres were perpendicular to the rotor surface. The built-up film on top of the fibres and on the matrix between the fibres was observed. The chemical composition of the film was studied by auger electron spectroscopy (AES). It is suggested that iron and copper oxides predominate at the sliding surface. The auger spectrum taken at a point on the film on top of the graphite fibre has the same characteristics as on the matrix between the fibres. Pearsall *et al.* [7] investigated the wear properties of unidirectionally oriented graphite-aluminium composites. SEM and AES observations showed that the low steady-state wear rate are due to formation of a film that impedes adhesion and confers some degree of self-lubrication. But their sliding speed varied between  $0.17$  and  $0.43 \text{ m s}^{-1}$  under a load of 4.46 N. The chemical composition of the composites pin the surfaces above a sliding speed of  $1 \text{ m s}^{-1}$  against the iron counter-face, and large amounts of oxygen probably combined with aluminium in the form of  $\text{Al}_2\text{O}_3$ . But no adhesion of iron to the pin was detected during time period. Alpas *et al.* [8] studied silicon carbide particulate-reinforced aluminium composites, whose worn surfaces showed that low wear rates were characterized by the formation of iron-rich transfer layers. The presence of iron in the deposited layer was demonstrated by energy dispersive spectroscopy (EDX) performed on the worn surfaces of the composites.

Comparison of the XPS results from worn surface samples showed that the amount of aluminium decreased in the N orientation at low speed in comparison to that for the N, P orientations at high speed conditions. This might be due to formation of a tribofilm on the N orientation sample at low speed. As a result of this, little fibre breakage or pulling-out of fibres occurred, and matrix removal from the composite pin during sliding was small because of the composites. This consisted mainly of continuous silicon carbide coated boron fibre and aluminium alloy matrix. However, in the high speed tests the situation was different because the amount of aluminium increased sharply and that of boron decreased greatly. This showed an opposite effect compared with the previous sample. It means that more fibre breakage took place in both N and P orientations at this speed and that some of the pieces were removed, while the rest of it formed a mechanically mixed alloy due to the large sliding speed. Thus, the amount of aluminium alloy increased considerably because of removal of the fibres from their place, and the carbon increased slightly due to atmospheric or surface contamination. However, for the P orientation, despite the fact that the same test conditions were used, the amount of iron was found to be lower than in the N orientation sample, although the aluminium alloy matrix had a similar content in both oriented composites. This could be due to differences in surface topography.

Comparing these results with the data in Table I, it is clear that the amount of oxygen in the surface is more or less independent of the wear conditions and orientations. However, the boron content changed appreciably: more boron in the N orientation, low speed, high load samples and also most of this extra boron in the form of an intermetallic compound, FeB<sub>2</sub>. The situation in the P orientation sample is less clear; the amounts of iron, despite the low load, is higher than in the low load N orientation sample according to the graph, but much lower according to the analysis of Table I. The reason for this is not known.

#### 4. Conclusions

From the experimental investigation of the wear properties of boron–aluminium composites under dry conditions, the following conclusions can be drawn:

(1) The MMCs showed excellent wear resistance compared with the unreinforced matrix when tested in air against a steel counter-face. The orientation of the fibres with respect to the sliding surface and direction had little effect on the wear rates. The N orientation gave the lowest wear at low speed and the P orientation slightly higher than the N orientation, but the trend changed when the tests were conducted at high speed.

(2) The XPS analysis showed that there was no significant variation in the amount of oxide over the surface under the test conditions used. At low speed in the N orientation, boron oxide and iron boride were formed, while the proportions of these components were different at high speed. This suggested that high speed led to mechanically mixed or alloyed material, with more iron boride and aluminium, but less boron in total, while low speed caused a mild form of wear in the N orientation.

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