# Ni-ZrB2 ELECTROCOMPOSITES.

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New generation materials require high temperature oxidation resistance besides many other tribological properties. Ni-ZrB<sub>2</sub> composites and their properties are dealt with in his paper.

Key words: Ni-ZrB2 composite, wear resistance, coating, oxidation resistance, electro composite.

#### INTRODUCTION

Use of metal ceramic composites for applications under extreme conditions of temperature, pressure and wear is well known. Cermets of various types have been reported [1, 2] to have many attractive properties such as enhanced creep resistance, superior wear and abrasion resistance and oxidation resistance. Electro-deposited composites are gaining importance because of the advantages such as low cost, ease and simplicity in producing thin, adherent and fairly uniform layers on various components, tailormade to the growing technological needs [3] Composites containing oxide dispersions are known to provide better oxidation resistance in air and at high temperature with moderate wear resistance while those containing carbides provide better wear resistance and moderate oxidation resistance. Mention has been made in the literature that Ni-ZrB2 composites possess high oxidation resistance and wear resistance. This paper discusses some of the important properties of electrodeposited Ni-ZrB2 composites [4]

### **EXPERIMENTAL**

The composites were produced form a Watt's bath of composition and operating conditions as follows: Nickel sulphate hexahydrate:  $250~\rm g \cdot l^{-1}$ ; nickel chloride hexahydrate:  $30~\rm g \cdot l^{-1}$ ; boric acid:  $40~\rm g \cdot l^{-1}$ . The anode was rolled nickel and the current density was  $450~\rm A \cdot m^{-2}$ . pH was maintained at 4 while the temperature was  $333~\pm~2k$ 

An addition of  $0.1~\rm g \cdot l^{-1}$  of sodium lauryl sulphate was made to prevent pitting as well as to ensure uniform dispersion of the particles in the deposit [5, 6]. Zirconium diboride powder of  $8-10~\mu m$  size was blended in mortar with small quantity of the electrolyte and transferred to the bulk solution in small increments to prevent agglomeration of particles in the electrolyte. Finally, the solution pH was adjusted electrometrically.

The electrolyte was maintained at 333  $\pm$  2K thermostatically and the solution was stirred using a mechanical stirrer (350 rpm).

Deposits plated on etched stainless steel were stripped in 1:1 nitric acid (v/v) and the resulting mixtures analysed to estimate nickel and the particulate matter [7]. Deposits plated on stainless steel up to a thickness of  $100-150~\mu m$  were used for evaluation of microhardness and abrasion resistance. Electroformed composites were also used for determining oxidation resistance.

Microhardness of the deposits was determined with knoop indentor at 50 g load. Resistance to abrasion was tested by making up of Taber Abraser at a load of 1 kg and a cs 10 calibrase wheel. The test has been carried out on each specimen for 2 cycles of 1000 revolutions and the wear index is the average of them.

Hardness measurement was done, after heat treatment of the specimens in a hydrogen atmosphere for 1 hour at different temperatures ranging from 473 to 1073 K [5].

Oxidation resistance of the specimens was assessed by heating the weighed ones in a current of air at two different temperatures viz. 973K and 1073K for one hour. The gain in mass due to oxide formation was taken as a measure of its resistance to oxidation.

The specimens were then analysed by X-ray diffraction with Cu K  $\alpha$  radiation before and after heat treatment. The structures of some of the composites were examined under a Scanning Electron Microscope before and after heat treatment.

# RESULTS AND DISCUSSION

The volume percent of zirconium diboride in the deposit increased as its concentration in the electrolyte was increased from 10 to 50 g·l<sup>-1</sup>. This trend is similar to that for silicon carbide incorporation with which the extent of codeposition is very high, and further incorporation becomes more difficult, because of excessive roughness of the deposits. With increasing current density, the particle incorporation was found to increase unlike in the case of alumina and TiO2 inclusion wherein the volume percent attains a steady value above 4 A·dm<sup>-2</sup>. As in the case of Ni-SiC system, the variation of  $\alpha \stackrel{i}{c}$  values where  $\alpha$  is the volume percent of particle in a deposit, i is the current density and c is the particle concentration in the electrolyte, shows linear increase with current density, indicating that the process is controlled by the strong adsorption of the particles [8]. This should be more due to the contribution from the conducting nature of the particles than the electrophoretic transport as in the case of inert particles. Table I shows the volume percent of ZrB2 obtained at different bath loadings.

TABLE-I: Effect of concentration of ZrB<sub>2</sub> particles in solution of the percentage incorporation in deposit.

Mass of particles in solution $(g \cdot l^{-1})$	Volume percent in solution (%)	Volume percent in deposit (%)
10	0.16	9
25	0.4	12
50	0.8	15

Table II shows the hardness and wear index of  $Ni-ZrB_2$  composites which is of the order of 500–550 KHN as against 250–265 KHN for pure nickel. The wear index of the composite

is found to be around 26–28 whereas it is 45.5 for pure nickel. On heat treatment at 1073K, the hardness was found to decrease to 400 KHN. (Table III) This hardness is, however, nearly 4 times higher than that for similarly heat treated nickel.

TABLE-II: Hardness and wear resistance of different composites.

**************************************	V V SAME SAME	Wear index	
System	Hardness	wear index	
	(KHN)	(2 cycles)	
Nickel	265	45.5	
Ni-SiC (7.3%)	500	30.5	
Ni-WC (15%)	450	30.0	
M-ZrB <sub>2</sub> (14.5%)	520	26.8	

TABLE-III: Effect of heat treatment on the hardness of the composites at different annealing temperatures

System	Hardness (KHN)		
	623K	873K	1073K
NI	200	175	100
Ni ZrB <sub>2</sub> (14.5%)	461	414	392

SEM micrographs of the wear tracks (Fig. 1) obtained with nickel and Ni-ZrB<sub>2</sub> composite show the higher wear resistance of the latter than the former.

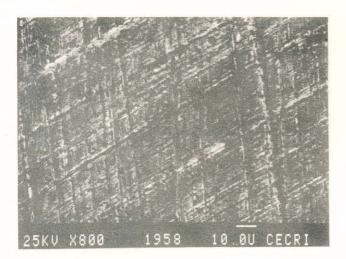


Fig. 1(a): SEM micrograph of the wear track on nickel deposit after 2 cycles

The XRD patterns of Ni-ZrB $_2$  composite as shown in Fig. 2b confirm that the peaks corresponding to ZrB $_2$  get broadened owing to microcrystallinity. A similar behaviour along with shifting of the peaks towards lower angles for nickel also confirms that there is a reduction in crystalline size. This is in conformity with the SEM micrographs (Fig. 3). Gain in mass on oxidation for Ni-ZrB $_2$  composite is found to be much less than for the oxide composites (Table IV).

Hence this composite appears to be useful where both high temperature hardness and oxidation resistance required.



Fig. 1(b): SEM micrograph of the wear track on Ni-ZrB $_2$  composite after 2 cycles

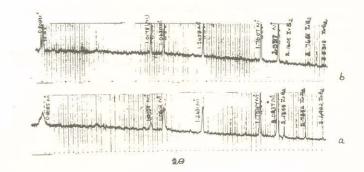


Fig. 2: X-ray diffractogram of Ni-ZrB<sub>2</sub> composite (a). X-ray diffractogram of Ni-ZrB<sub>2</sub> composite after annealing at 1073k(b).

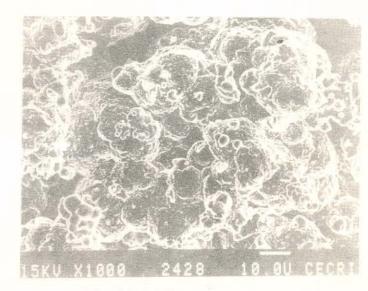


Fig. 3(a): SEM micrograph of Ni-Zrb2 composite

# CONCLUSION

Ni-ZrB<sub>2</sub> composites posses hardness of high order along with very good oxidation resistance.



Fig. 3(b): SEM micrograph of Ni-ZrB2 composite after annealing at 1073k.

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TABLE-IV: Oxidation Resistance of Various Electrocomposites

System	TEMPERATURE (K)		
		eight gain ng/dm <sup>-2</sup>	
	937K	1073K	
Nickel	25.0	42.2	
Ni-TiO <sub>2</sub> (7%)	14.07	20.05	
Ni-Al <sub>2</sub> O <sub>3</sub> (5.6%)	15.2	20.1	
Ni-ZrB <sub>2</sub> (15%)	10.1	18.2	

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