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Separation of ceramic particles from aluminium metal–ceramic composites

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An improved method for separation and quantitative analysis of ceramic particles in aluminium–ceramic mixture and cast materials is given. The mixture containing H_2SO_4, HNO_3 and HCl has been found to be most efficient in the separation process. Mixtures containing other acids have been shown to be unsuitable. Optical and scanning electron micrographs of acid-treated aluminium–graphite and talc samples are presented.

1. Introduction

Improvement in lubrication and wear resistance properties of aluminium-alloy is essentially dependent upon the volume and extent of homogeneity of ceramic particles present in the aluminium–ceramic composite material [1–4]. The latter is usually made following a casting process where the ceramic particles are dispersed in a molten metal matrix. The casting process very often allows the escape of some ceramic particles during heat treatment causing uncertainty in mass balance; the percentage of ceramic particles added does not remain the same as in the cast materials. Therefore, chemical analysis of the end product is absolutely essential. Although methods of analysis, dissolution or separation of various metal ions are well known for aluminium metal and alloys [5–7], there is practically no report available on the separation or analysis of ceramic particles in aluminium–ceramic composite materials. Even for analysis of aluminium and aluminium-alloy, different acid media have been used, and confusion exists as to which mixture should be selected. The only work in this regard is by Burke and Albright [8]. According to these authors, gravimetric estimation takes five minutes which is quite surprising.

The first step needed in chemical or microscopic analysis is the separation of ceramic particles from the composite [9]. Any separation procedure adopted should be such that the ceramic particles can be recovered without destruction. This is especially applicable to the layered minerals which are often used as additives in the preparation of the composite [1–4].

2. Experimental details

Six acid mixtures were used which were tested for both known dry mixture and cast composite samples. Table I shows the amount of acids and water used. The first four mixtures are selected because HNO_3 and $HClO_4$ are strong oxidizing agents, and the fifth mixture is aqua regia which is already well known as an oxidizing agent. The sixth acid, HCl, is selected because it is the only reducing acid. The graphite used for the mixture is synthetic (−45 μm).

Acid treatment will liberate silicon metal and probably traces of silicic acid from the alloy. These will be retained in the residue along with the ceramic particles. Traces of metal ions will also adhere to the residue. In order to separate the pure ceramic particles, the residue is treated with 25 to 30% NaOH solution followed by addition of HNO_3 + HCl mixture

TABLE I Analysis of aluminium–ceramic materials in different acid mixtures

Acid mixture	% ceramic particles in known mixture							
	Graphite		Talc		Mica		Zircon sand	
	Added	Obtained	Added	Obtained	Added	Obtained	Added	Obtained
1. $H_2SO_4 + HNO_3 + HCl + H_2O$ (6.3)*(10)(10)(23.7)	3.85	3.84	5.67	5.64	5.48	5.61	18.79	18.96
2. $HClO_4 + HNO_3 + HCl + H_2O$ (12.5)(10)(10)(17.5)	3.87	3.84	–	–	–	–	–	–
3. $HClO_4 + H_2SO_4 + H_2O$ (4.3)(31.2)(14.5)	2.85	3.83	–	–	–	–	–	–
4. $HClO_4 + HNO_3$ (33.3)(16.7)	1.09	4.04	–	–	–	–	–	–
5. HNO_3 + HCl (12.9)(37.5)	4.37	4.08	–	–	–	–	–	–
6. HCl	4.07	3.92	–	–	–	–	–	–

*Values in parenthesis indicate volume in ml.

TABLE II Analysis of ceramic particles in LM-13 ceramic cast materials

% graphite added	% graphite retained in cast material	% talc added	% talc retained in cast material	% zircon sand added	% zircon sand retained in cast material
3.0	1.21	3.0	0.48	25.0	23.0
5.0	4.41	4.0	0.90	30.0	25.7
7.5	5.13	4.5	2.40	40.0	31.6

(1 : 2). This will facilitate filtration of monomeric silicic acid. The residue is washed successively with (a) water, (b) NaOH (5%), (c) water, (d) HCl (5%) and (e) finally with water until the residue is free from acid.

3. Results and discussion

The likely chemical reactions are as follows:

$$\begin{tikzcd} \mathrm{Si^0_{alloy}} \quad + & \text{acid mixture} \arrow[r] & \mathrm{Si^0 + (H_2SiO_3)} \arrow[d, "\mathrm{HNO_3 + HCl + NaOH}"] \\ \mathrm{SiO_2} & \mathrm{H_2SiO_3} \ \text{(monomeric silicic acid).} \arrow[l, "\text{drying}"'] & \mathrm{Na_2SiO_3} \arrow[l] \end{tikzcd}$$

The above procedure is perfectly valid for aluminium–graphite, aluminium–talc, aluminium–mica and aluminium–zircon composite materials. But the problem of separation becomes complicated when ceramic materials containing free silica such as sand and rice husk ash are taken as additive in products of cast metal–ceramic composites. The free silica has a tendency to react with molten aluminium during the casting process forming $Al_2(SiO_3)_3$ in the following way:

$$x\mathrm{Al/Al_2O_3} + y\mathrm{SiO_2} \rightarrow \mathrm{Al}_x(\mathrm{SiO_3})_y$$

Formation of aluminium silicate depends upon the various thermodynamic parameters involved in the casting process. Consequently, the final cast product will have SiO_2, and complexes of aluminium with SiO_2. SiO_2 in sand is in the crystalline state whereas it is amorphous in rice husk ash, as a result of which the latter will be highly reactive compared to sand. The final cast product will contain silicon, SiO_2 and $Al_x(SiO_3)_y$, converted to Na_2SiO_3, as in the case of other aluminium–ceramic composites.

Percentages of ceramic particles obtained in mixtures containing a known amount of LM-13 chips along with graphite, talc, mica powder ($-45\,\mu$m) zircon sand and cast ceramic materials are tabulated in Tables I and II, respectively. While LM-13 graphite mixtures have been treated in all acid mixtures, LM-13 + talc, LM-13 + mica and LM-13 + zircon sand have been tested only in the acid solution of $H_2SO_4 + HNO_3 + HCl$. The results show that percentages of ceramic particles added and obtained are fairly close.

The results indicate that the mixtures of $H_2SO_4 + HNO_3 + HCl$ and $HClO_4 + HNO_3 + HCl$ are best. The quantities of HNO_3 and HCl used do not correspond to that of aqua regia. However, addition of a third acid to the mixture such as H_2SO_4 or $HClO_4$ make the mixture very effective. Mixtures having two acids, ($HClO_4 + HNO_3$ or $H_2SO_4 + HClO_4$, surprisingly do not give accurate results.

Table II indicates that in the cast composite materials, 100% ceramic particles cannot be retained. This is due to (1) density difference between metal matrix and ceramic particles, (2) wettability of ceramic particles in the matrix, and (3) casting parameters, e.g. temperature, degassing time, stirring process, etc. Of the above mentioned ceramic particles, talc has the least retention property.

Time taken for acid treatment of aluminium metal–ceramic composite is also important. The separation

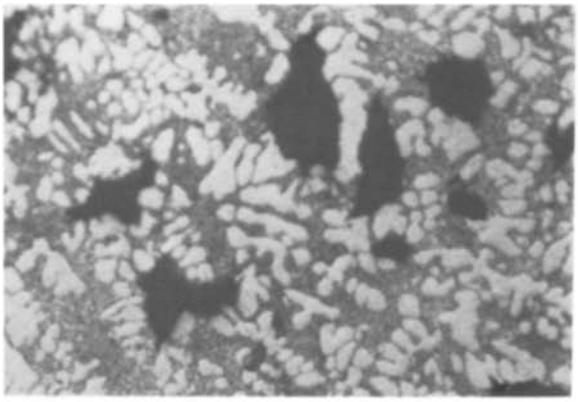

Figure 1 Optical micrograph of LM-13 graphite cast composite (× 140).

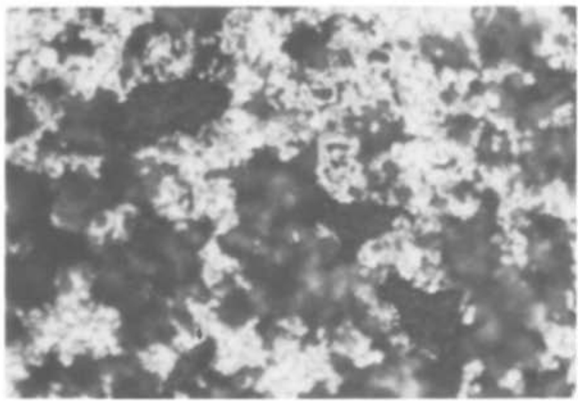

Figure 2 Optical micrograph of LM-13 graphite cast composite treated with acid mixture 1, for 1 min (× 140).

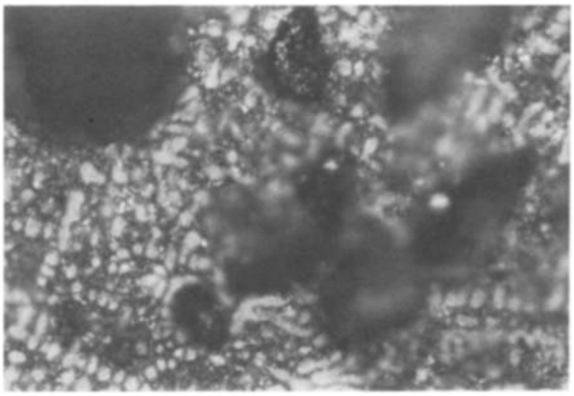

Figure 3 Optical micrograph of LM-13 graphite cast composite treated with acid mixture 1, for 3 min (× 210).

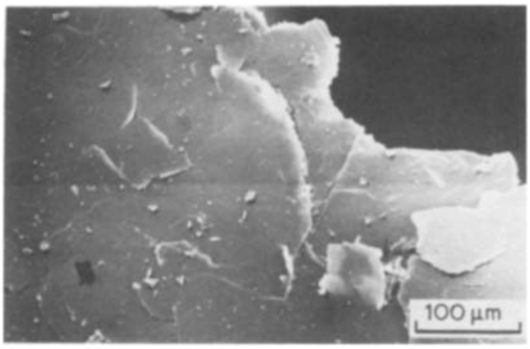

Figure 4 Scanning electron micrograph of talc sample.

of ceramic particles remains incomplete if the time of dissolution is less.

Beneficiation of ceramic raw materials excepting graphite, is very important because the impurities present will lead to erroneous results. The naturally occurring minerals, on treatment with acid mixture and alkali, have been found to give an impurity level of 2 to 6%. These impurities are clays and other acid-soluble materials [9].

Effect of acid treatment on the cast composite has been studied by optical and scanning electron microscopy. Figs 1 to 3 show successively that the graphite particles gradually become separated from the aluminium matrix on acid treatment. Complete separation of graphite from the matrix is accomplished within 20 to 30 min.

The particles separated by the above treatments have been examined by optical and scanning electron microscopy. The results indicate that the topographical features of the ceramic particles remain unaffected. Fig. 4 shows a scanning electron micrograph of talc, separated by acid treatment.

The present method has many advantages over that of Burke and Albright [8] apart from the fact that the method is applicable to metal–ceramic composites containing mica and talc including graphite. A simple filtration technique can be applied here, unlike the glass fibre filter described by those authors. 25 to 30% NaOH solution is adequate for dissolution of the sample here. As heating in a furnace can be avoided by the present method, there is also a considerable saving in time.

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