JJMIE

Volume 8 Number 5, October 2014
ISSN 1995-6665
Pages 313 - 321

Jordan Journal of Mechanical and Industrial Engineering

Fabrication and Characterization of Copper-Red Mud Particulate Composites Prepared by Powder Metallurgy Technique

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Received 17 Jul 2014 *Accepted 10 Sep 2014*

Abstract

Copper-red mud powder mixtures containing 0-14 wt% red mud with 75, 53 and 38 μm particle sizes were prepared. Small cylindrical specimens of 9 mm diameter and 10.5 mm length were fabricated at 300 MPa, using single action die compaction at ambient temperature. These compacts were sintered in argon atmosphere at 8500C. The physical, mechanical and electrical properties of green and sintered compacts were determined as a function of particle size of red mud and its weight percent. It was observed that the ejection pressure, green density, and strength decreased, while spring back, green porosity and hardness increased with increasing the weight percent of red mud. It was also observed that ejection pressure, green density, green hardness and strength increased, while spring back and green porosity decreased with decreasing particle size of red mud. Sintering resulted in a decrease in volume and an increase in the density of green compacts. It was found that the sintered density of the compacts decreases with increasing weight percent of red mud. It was also observed that the addition of red mud results in an increase in hardness and a decrease in compressive yield strength and electrical conductivity of sintered compacts under the present experimental conditions. It was further observed that with the decrease in particle size of red mud, the density, hardness, compressive yield strength and electrical conductivity of sintered compacts gradually increase.

Keywords: *: Ejection Pressure, Spring Back, Density, Porosity, Hardness, Strength, Electrical Conductivity*.

1. Introduction

Management of waste materials has become very much important for protecting the environment. One way to achieve this objective is to reuse/recycle the waste materials for the development of new materials. The alumina manufacturing industry produces voluminous quantity of red mud as a waste product. It is estimated that an amount of 90 million tons of red mud is produced annually in various parts of the world. Its disposal causes significant economic and environmental problems [1-3]. As a result, exploring new avenues to utilize red mud is highly essential. To solve the disposal problem, considerable research and development work have been carried out, and efforts are made to use red mud as a cheap adsorbent for removal of toxic metals such as nickel [4], arsenic [5], lead and chromium [6] from aqueous solutions, for preparation of building materials, namely iron rich cement and ceramic tiles [7, 8], and as a filler material in metal and polymer matrix composites [9-13].

Copper matrix composites with particulate ceramic reinforcements such as SiC and Cu_2O are potential candidate materials for electrical and electronic applications [14-18]. These copper composites combine the superior ductility, toughness and thermal conductivity of copper and high strength, improved wear resistance and low coefficient of thermal expansion of these ceramic reinforcements. However, the high cost of these ceramic reinforcements remains a major barrier in their wide spread use. Hence, there has been an increasing interest in composites containing low density and low cost reinforcements such as fly ash [19] and red mud.

Red mud mainly consists of silica and oxides of aluminum, iron, and titanium, along with other minor constituents. Red mud particles have been used as filler in aluminum and as well as polymer matrices. It is reported that the addition of red mud to aluminum matrix increased the hardness and wear resistance and decreased the coefficient of friction [9, 10]**.** The addition of red mud to polymer matrices has increased tensile strength, impact and flexural strengths considerably [11-13]. Hence, this low cost and low density particulate reinforcement is added to copper matrix to study the effect of red mud content and its particle size on the physical, mechanical and electrical properties of the copper-red mud composites for possible electrical and electronic applications.

Several processing techniques are used for the production of the metal matrix composites, which can be grouped into two main routes depending on the state of the matrix during the fabrication process, either liquid or solid routes. Copper-red mud composites by casting are likely to

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exhibit segregation and non-uniform distribution of particles because of the differences in density between the red mud particles and the matrix. So, powder metallurgy is used to prepare copper-red mud composites in the present work. With this aim, the present experimental investigations are carried out on fabrication and characterization of copper-red mud particulate composites prepared by powder metallurgy technique.

2. Experimental Procedure

Copper powder (99.5% pure) was procured from M/s Loba Chemie Pvt. Ltd., Mumbai and the red mud material was obtained from the aluminum refinery of NALCO located at Damanjodi, Orissa, India. The average size of copper powder is 44 µm. The chemical analysis of the red mud was carried out by M/s Natural Resource Development Cooperative Society Ltd., Hyderabad, India. The chemical composition of red mud is shown in Table 1.

The coarse particles of red mud, obtained from the aluminium refinery, were pulverized into finer particles manually. In order to study the effect of red mud particle size on the properties of copper-red mud composites, three different sizes of red mud powders, namely 75, 53 and 38 µm were prepared using sieve analysis. The densities of these red mud powders were measured using Archimedes principle and are represented in Table 2. Mixtures of copper-red mud powders containing 0-14 wt % red mud with 75, 53 and 38 µm particle sizes were prepared. In order to obtain uniform distribution of copper and red mud powders, they were mixed mechanically using a rotating rectangular container for a period of one hour. Cylindrical compacts were obtained at 300 MPa using single action die compaction at ambient temperature. The specimens were compacted at a uniform load rate of 5 kN/min for a period of 3.8 min. Silicone spray was used as the die wall lubricant. The compact dimensions were 9 mm diameter and 10.5 mm length. The above compacts were sealed in transparent silica tube under argon atmosphere and sintered at 850^0C in a tubular furnace for a period of 45 min.

Scanning electron micrographs were used to study the structural details of the particles. Metallographic examination of green and sintered compacts was carried out using optical microscopy. The green properties, namely spring back, ejection pressure, green density, green porosity, green hardness and green strength were evaluated as a function of particle size of red mud and its weight percent. Density, porosity, hardness, compressive yield strength and electrical conductivity of the sintered compacts were also determined as a function of particle size of red mud and its weight percent. The spring back of the cylindrical green specimen is determined using the following equation:

$\%\ Spring\ back = (D_g - D_d) \times 100/D_d$,

where

D_g = *Diameter of the green specimen*

D_d = *Diameter of the die bore (9 mm)*

Green and sintered densities were determined by physical measurements. The porosity of the green and sintered compacts was determined by taking theoretical density of the specimen into consideration. Vickers hardness measurements were obtained using TIME TH130 Integrated Micro Hardness Tester. Compression testing was conducted using an electronic UTM at a crosshead speed of 0.2 mm/min. The electrical conductivity was measured in % IACS using a digital electrical conductivity meter.

Table 2. Density of red mud

Particle size of Red mud	Density (kN/m^3)
0-75 microns	29.23
0-53 microns	29.42
0-38 microns	30.35

3. Results and Discussion

3.1. Powder Characteristics

The scanning electron micrographs of copper and red mud particles as shown in Figures 1 & 2 indicate their size, shape, size distribution and structure. Figure 1 indicates the flaky and dendritic structure of copper powder, whereas Figure 2 reveals the presence of partially round, angular and elongated shapes of red mud particles.

3.2. Green Properties

The variation of spring back with increasing red mud weight percent and its particle size is shown in Table 3 as well as Figure 3. They show that spring back increases with the increase in red mud content. It is known that spring back increases with increasing plastic strain and yield stress and decreasing modulus of elasticity of the particles. The relative increase in spring back with the addition of red mud is due to the deformation of red mud particles under elastic mode during compaction. It also shows that spring back decreases with decrease in particle size of red mud. This may be attributed to the red mud particle size distribution.

The variation of ejection pressure with increasing red mud content and its particle size is presented in Table 3 as well as Figure 4. They indicate that the ejection pressure decreases with the increase in red mud content. The high ejection pressure in the case of pure copper compacts is due to the high frictional characteristics of flaky and dendritic copper powder. The gradual decrease in ejection pressure with increasing red mud weight percent may be attributed to the low frictional properties of partially rounded red mud particles in the composite. The increase in ejection pressure with decrease in particle size of red mud may be due to increase in real area of contact between the die wall and the green compact. The optical microstructure of copper-red mud green compact is shown in Figure 5. The figure shows the deformation and uniform distribution of 6 wt% of 38 µm red mud powder in copper matrix at 300 MPa compaction pressure.

Table 1. Chemical composition of red mud (wt%)

Constituents	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	CaO	Na_2O	P_2O_5	V_2O_5	ZnO	MgO	MnO	K_2O
Wt%	14.14	11.53	48.5	5.42	3.96	7.5	0.3	0.12	0.03	0.05	0.17	0.06

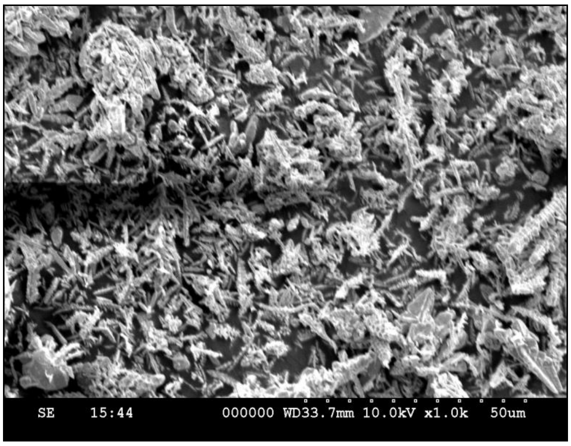

Figure 1. SEM of copper particles

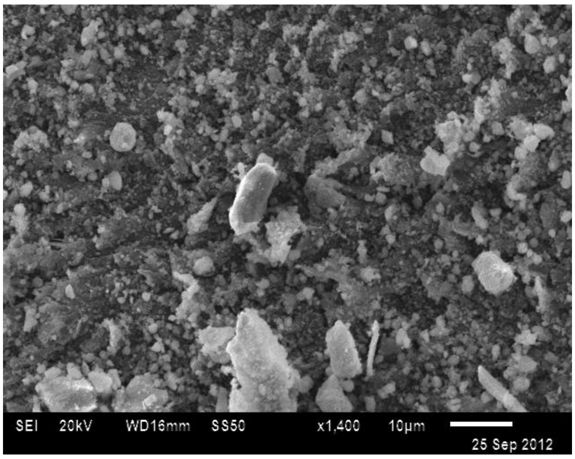

Figure 2. SEM of red mud particles (38 μm)

Table 3. Effect of red mud content and particle size on spring back & ejection pressure

Composition	Spring back (%)			Ejection Pressure (MPa)		
	Size of Red mud particles			Size of Red mud Particles		
	75 µm	53 µm	38 µm	75 µm	53 µm	38 µm
Copper	0.446			32		
Copper + 2% Red mud	0.505	0.498	0.459	18.46	22.46	29
Copper + 4% Red mud	0.522	0.502	0.476	16.8	21.04	26.93
Copper + 6% Red mud	0.537	0.516	0.503	16.01	19.83	26.23
Copper + 8% Red mud	0.544	0.525	0.519	15.89	18.05	23.56
Copper + 10% Red mud	0.551	0.539	0.53	14.05	17.56	21.31
Copper + 12% Red mud	0.631	0.572	0.56	12.83	15.62	18.54
Copper + 14% Red mud	0.637	0.625	0.602	11.7	13.71	17.71

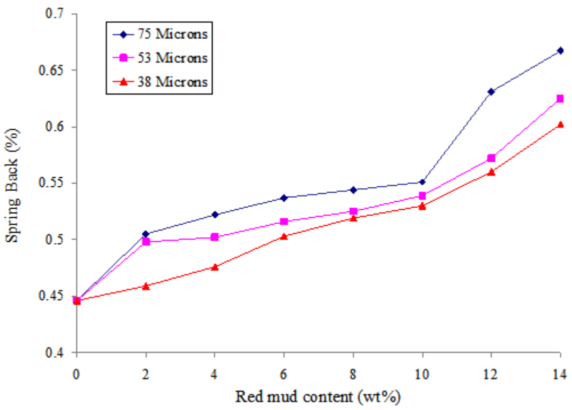

Figure 3. Effect of red mud content on spring back

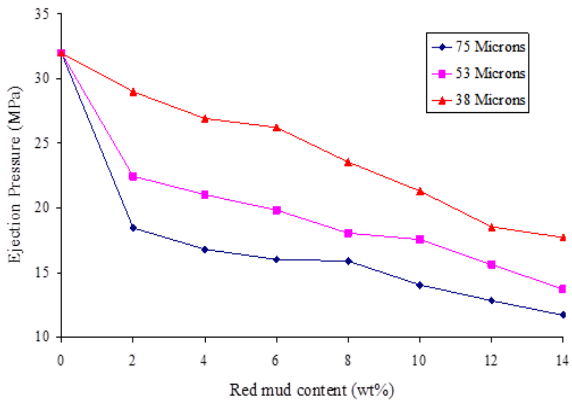

Figure 4. Effect of red mud content on ejection pressure

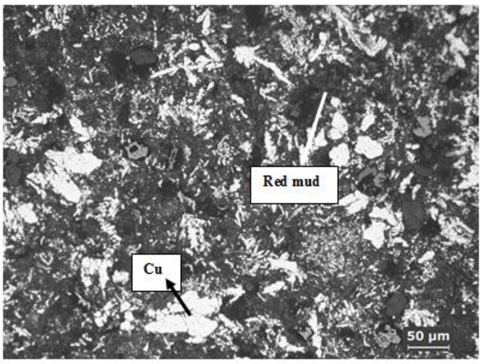

Figure 5. Microstructure of Cu-6% Red mud (38 µm) green compact

The effect of red mud weight percent and its particle size on green density and porosity are shown in Tables 4 & 5 and Figures 6 & 7, respectively. They show that green density decreases, whereas porosity increases with increase in red mud weight percent. The decrease in green density with addition of red mud to copper matrix is due to the relative density differences of red mud and copper powder mixtures. The increase in porosity with the addition of red mud can be attributed to the partially round shape of red mud particles. It also shows that green density increases while green porosity decreases with decrease in particle size of red mud. Finer particles of red mud result in more particle to particle contacts and plastic deformation leading to cold welding of particles. This results in more density and less porosity of the green compacts. Larger particles of red mud may lead to the partial welding of particles and voids resulting in less density and more porosity of the green compacts.

Table 4. Effect of red mud content and particle size on density

Composition	Green Density (kN/m^3)			Sintered Density (kN/m^3)		
	Size of Red mud particles			Size of Red mud Particles		
	75 µm	53 µm	38 µm	75 µm	53 µm	38 µm
Copper	68.26			77.97		
Copper + 2% Red mud	58.83	62.02	66.68	69.02	70.4	72.05
Copper + 4% Red mud	54.35	60.69	63.91	63.57	67.71	70.71
Copper + 6% Red mud	54.2	57.98	61.18	62.96	64.03	66.02
Copper + 8% Red mud	54.05	56.59	59.99	60.09	60.07	63.78
Copper + 10% Red mud	53.6	54.87	57.58	58.18	58.58	60.32
Copper + 12% Red mud	47.23	51.1	54.03	50.43	55.36	56.33
Copper + 14% Red mud	45.48	51.12	53.26	48.46	53.22	54.69

Table 5. Effect of red mud content and particle size on porosity

Composition	Green Porosity (%)			Sintered Porosity (%)		
	Size of Red mud particles			Size of Red mud Particles		
	75 µm	53 µm	38 µm	75 µm	53 µm	38 µm
Copper	18			12.01		
Copper + 2% Red mud	26.25	22.59	21.25	18.3	16.56	15.02
Copper + 4% Red mud	30.32	26.31	21.73	20.34	16.66	15.98
Copper + 6% Red mud	30.88	27.45	22.35	20.87	18.24	16.31
Copper + 8% Red mud	31.02	27.23	21.8	21.16	19.87	16.38
Copper + 10% Red mud	33.16	27.68	22.37	22.15	20.85	18.41
Copper + 12% Red mud	33.99	28	24.48	28.84	21.76	21.25
Copper + 14% Red mud	34.08	28.63	24.92	29.41	22.33	21.37

Table 6. Effect of red mud content and particle size on hardness

Composition	Green Hardness (HV)			Sintered Hardness (HV)		
	Size of Red mud particles			Size of Red mud Particles		
	75 µm	53 µm	38 µm	75 µm	53 µm	38 µm
Copper	30			24.6		
Copper + 2% Red mud	30.9	31.7	32.4	25.14	26.6	27.08
Copper + 4% Red mud	33.7	35.5	38.2	27.75	29.61	31.73
Copper + 6% Red mud	35.2	36.6	42.1	32.58	34.4	35.16
Copper + 8% Red mud	37.3	38.6	42.9	36.33	37.93	38.38
Copper + 10% Red mud	39.2	40.6	43.8	38.39	39.94	42.03
Copper + 12% Red mud	43.2	45.8	50.1	41.48	43.01	48.66
Copper + 14% Red mud	46.4	47.3	51.2	44.39	45.08	49.13

Table 7. Effect of red mud content and particle size on strength

Composition	Green Strength (MPa)			Sintered Compressive Yield Strength (MPa)		
	Size of Red mud particles			Size of Red mud Particles		
	75 µm	53 µm	38 µm	75 µm	53 µm	38 µm
Copper	152			56.6		
Copper + 2% Red mud	116.29	125.09	136.68	49.4	51.74	53.83
Copper + 4% Red mud	105.63	115.83	125.4	42.7	44.23	48.57
Copper + 6% Red mud	95.13	106.71	120.31	36.5	38.33	43.06
Copper + 8% Red mud	81.7	97.45	106.87	31.87	34.38	38.91
Copper + 10% Red mud	73.36	82.47	101.93	28.32	33.29	35.04
Copper + 12% Red mud	61.31	71.97	89.26	26.38	29.18	33.36
Copper + 14% Red mud	52.97	55.59	65.32	24.08	27.56	31.08

Tables 6 & 7 and Figures 8 & 9 represent the red mud content's effects and its particle size on green hardness and strength. They show that the green hardness increases, while the green strength decreases with increasing red mud content. The increase in green hardness with increase in red mud content is due to the presence of silica and oxides of aluminium, iron and titanium present in the red mud. The decrease in green strength with increasing red mud content may be attributed to the poor mechanical bonding between the red mud and the copper powder mixture. This may be because of the round surface and lack of plasticity of red mud particles as compared to that of copper. These figures also show that the green hardness and strength increase with the decrease in the particle size of red mud.

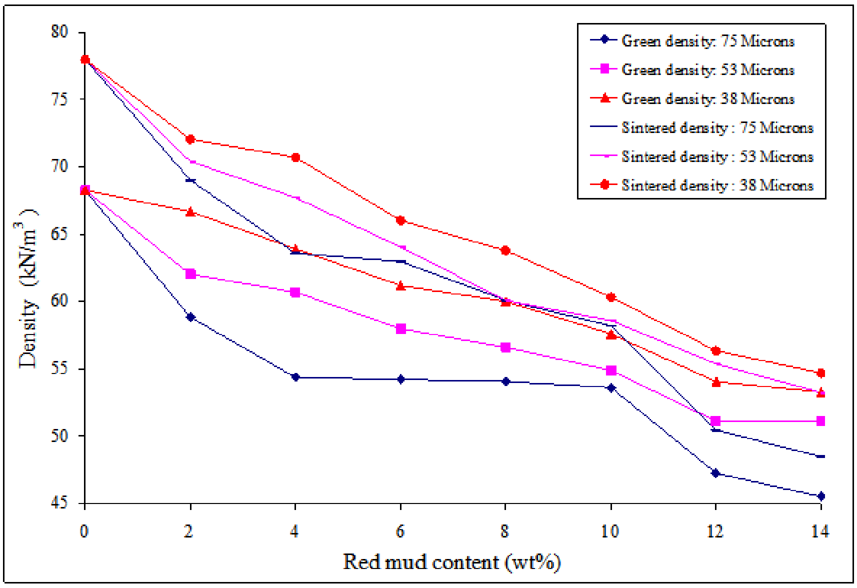

Figure 6. Effect of red mud content on density

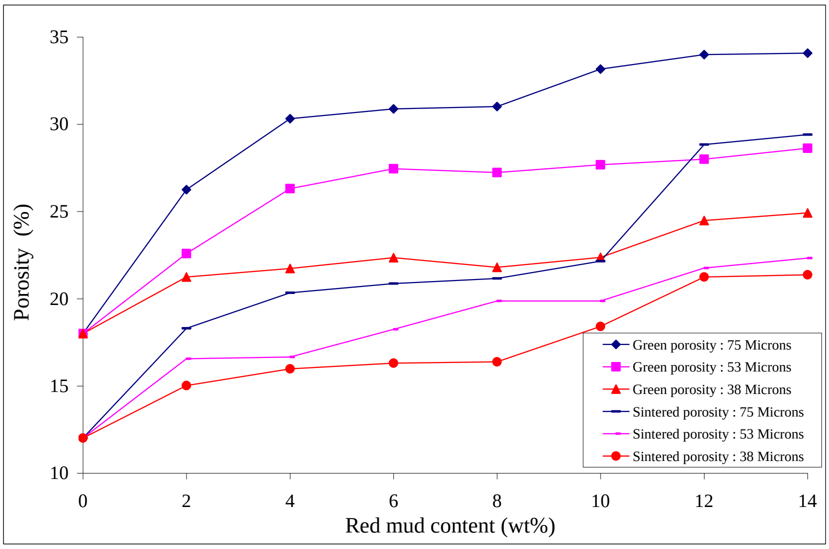

Figure 7. Effect of red mud content on porosity

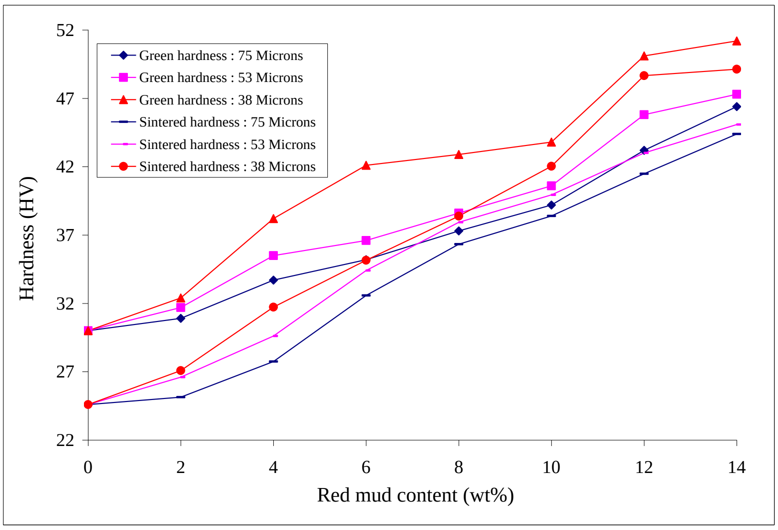

Figure 8. Effect of red mud content on hardness

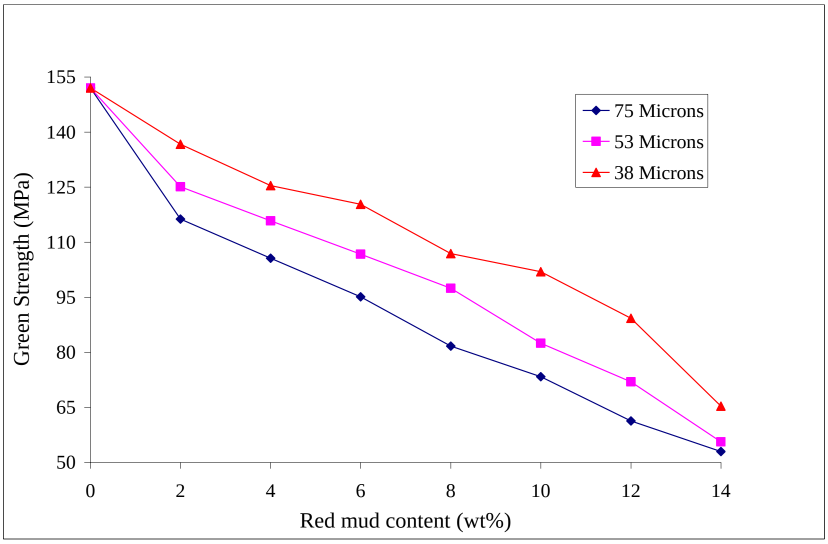

Figure 9. Effect of red mud content on green strength

4. Sintered Characteristics

Dimensional changes (volume changes) always occur during sintering of a green compact due to solid state diffusion processes or liquid phase sintering in a multi-component system with widely different melting points of constituents. Since the sintering temperature employed in the present investigation (850^0C) is less than the melting point of copper (1083^0C) and red mud (1300^0C)[3], the composites undergo solid state sintering.

Tables 4 & 5 and Figures 6 & 7 also indicate the red mud content's effect and its particle size on sintered density and porosity. Comparing the sintered densities with those of the corresponding green densities for respective compositions, it can be clearly noticed that the density after sintering is always more than the corresponding green density due to negative growth in volume on sintering. Sintered density decreases with increasing red mud content. Sintered porosity increases with increasing red mud content. The addition of 38 µm red mud powder to copper increased the sintered porosity of pure copper compacts from 12% to 21.3%. Since the density of red mud is very low, for a given weight percentage, significant volume of matrix phase is replaced. With increase in red mud content, the proportion of direct red mud-red mud contacts increases. The direct red mud-red mud contacts degrades the quality of sintering at the processing temperature, because the red mud has a melting point (1300^0C) higher than the sintering temperature. This aids in the reduction of volume change and sintered density with a corresponding increase in sintered porosity of the composites. Figures 6 & 7 also show that the sintered density increases with the corresponding reduction in sintered porosity with decrease in particle size of red mud. The microstructure of copper-6% red mud (38 µm) sintered compact depicted in Figure 10 shows a uniform dispersion of red mud in the copper matrix.

Tables 6 & 7 and Figures 8 & 11 represent the variation of sintered hardness and compressive yield strength of the copper-red mud composites as a function of red mud weight percent and its particle size. The figures show that sintered hardness increases, while sintered compressive yield strength decreases with increase in red mud content. These figures also show that with decrease in particle size, sintered hardness and yield strength increases. The sintered compressive yield strength is particularly low for composites containing more than 10 wt% red mud. It indicates that the useful range of red mud that can be added to copper matrix lies below 10%. The decrease in strength is due to the high porosity and ineffective sintering between red mud particles and the copper matrix in sintered red mud composites.

The red mud's effect and its particle size on electrical conductivity is shown in Table 8 and Figure 12. Electrical conductivity gradually decreases with the increase in red mud content. This may be due to the poor conductivity of the constituents of red mud apart from the low density and high porosity of the sintered red mud compacts. The electrical conductivity of the sintered red mud compacts gradually increases with decrease in particle size of red mud. This can be attributed to the increased density of compacts with decrease in particle size.

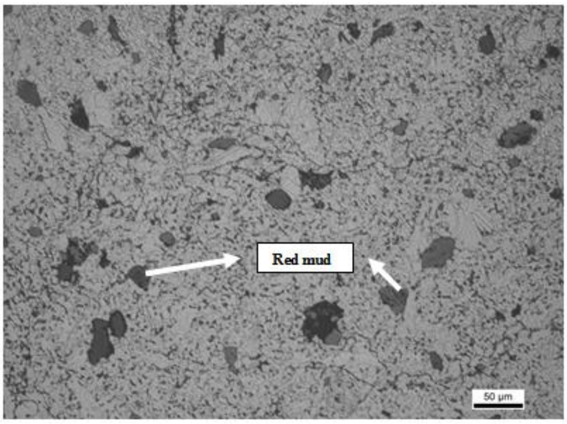

Figure 10. Microstructure of Cu-6% red mud (38 µm) sintered compact

Table 8. Effect of red mud content and particle size on electrical conductivity

Composition	Electrical Conductivity (% IACS)		
	Size of Red mud particles		
	75 µm	53 µm	38 µm
Copper	44.2		
Copper + 2% Red mud	41.7	42.6	43.5
Copper + 4% Red mud	39.3	40.8	42.3
Copper + 6% Red mud	34.6	38.8	41.2
Copper + 8% Red mud	32.5	33.2	33.6
Copper + 10% Red mud	25.2	28.5	29.8
Copper + 12% Red mud	23.7	25.8	27.6
Copper + 14% Red mud	20.9	22.4	24.1

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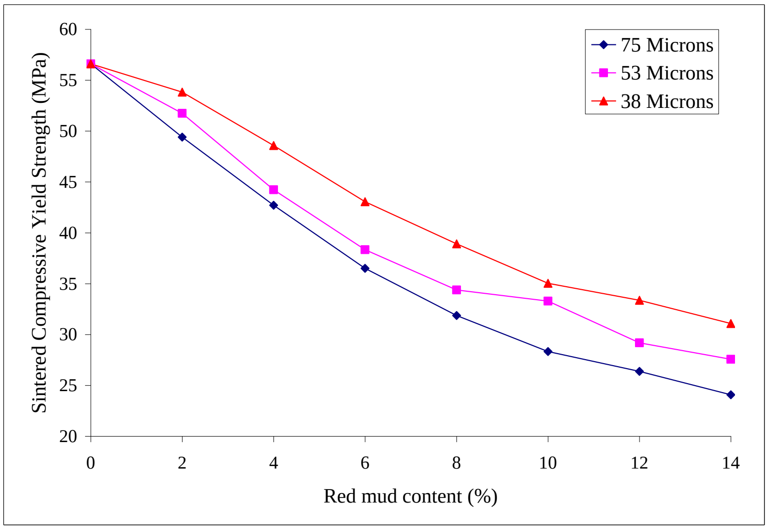

Figure 11. Effect of red mud content on sintered compressive yield strength

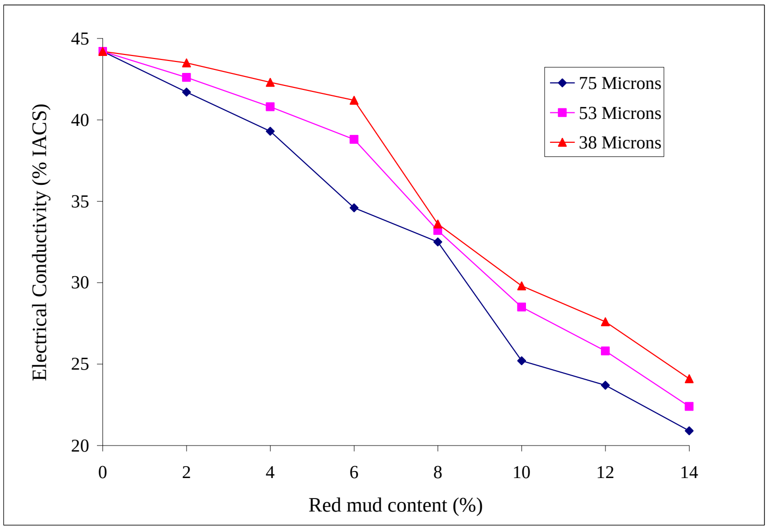

Figure 12. Effect of red mud content on electrical conductivity

Conclusions

From this investigation, the following conclusions are obtained:

The copper-red mud composites with uniform dispersion of red mud can be obtained by powder metallurgy processing route. Incorporation of red mud particles modified the physical, mechanical and electrical conductivity properties of the pure copper compacts. Ejection pressure, green density and strength decreased with increase in red mud content. Spring back, green porosity and hardness increased with increase in red mud content. Ejection pressure, green density, green hardness and strength increased with decrease in particle size of red mud. Spring back and green porosity decreased with decrease in particle size of red mud.

Sintering of copper-red mud compacts resulted in an increase in density of the green compacts and sintered density decreased with increasing red mud content. Sintered porosity and hardness increased with increasing red mud weight percent. Compressive yield strength of sintered copper-red mud composites decreased with increasing red mud weight percent. The electrical conductivity of the sintered copper compacts decreased with addition of red mud.

Sintered density, sintered hardness, sintered compressive yield strength and electrical conductivity gradually increased with decrease in particle size of red mud. Sintered porosity decreased with decrease in particle size of red mud. These results suggest that the useful range of red mud that can be added to copper lies below 10% and finer particles of red mud result in improved physical, mechanical and electrical conductivity properties of the copper- red mud composites.

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