

IMPROVEMENT IN HARDNESS AND CORROSION RESISTANCE OF STEEL THROUGH RED MUD COATING

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Abstract: Red mud is a waste product produced during alumina extraction from bauxite by the Bayer's process and has become a major issue in connection with its disposal. As the aluminum demand in the world is increasing, the disposal of red mud waste into the environment also increases, bringing with its environmental issues coupled, however, a potential for the waste to generate a huge amount of unutilized resources still contained in the red mud, such as Fe_2O_3 , Al_2O_3 , SiO_2 , and others. As a possible useful utilization of the disposable waste, red mud can be used as a fine ceramic coating material onto metal substrates. For a fine ceramic coating, fine red mud powder was mixed with other substances to form a non-aqueous coating slurry with toluene as the solvent, PEG 6000 as the binder and plasticizer, and sesame oil as the dispersant. The coating process was performed by a dip-coating method onto ST-37 steel with a withdrawal speed of 0.23 mm/sec. The densification of coating material took place after calcinations at 900 °C, 1100 °C, and 1300 °C for 2 hours in nitrogen atmosphere. This treatment evinced an improvement of the surface hardness from 146.2 HV to 1738.8 HV for the sample calcined at 1100 °C and the corrosion resistance by 4.14 times, for the sample calcined at 1300°C.

Keywords: Ceramic coating, Corrosion resistance, Hardness, Non-aqueous coating slurry, Red mud.

Introduction

The production of aluminum metal from bauxite commercially comprises of two processing steps, namely the extraction of alumina from bauxite by the Bayer's process [1] and the electrolyzation of alumina in a Hall-Heroult cell [2]. The waste by-product of the alumina extraction from bauxite is known as red mud. Through the Bayer's process, approximate 35-40% of the processed bauxite goes into red mud waste and becomes the biggest problem in alumina production because it generally exits the process stream as a highly alkaline slurry (pH 10-12.5) containing about 15-30% solids [3,4,5]. About 90 million metric tons of red mud are produced each year [6]. This huge amount of waste makes serious problems to the waste storage due to the need of large waste reservoirs and it will also impose an environmental impact. The red mud still contains important materials for industrial processes, such as Fe_2O_3 (30-60 wt%), Al_2O_3 (10-20 wt%), SiO_2 (3-50 wt%), Na_2O (2-10 wt%), CaO (2-8 wt%), and TiO_2 (0-25 wt%). Therefore, many researches had been conducted to utilize red mud for various applications, such as wastewater treatment for removing toxic metals [3,7,8], building materials [4,9], adsorbents, and catalysts [5].

Although many researches and developments have been conducted on the utilization of red mud, there were almost no attempts on the utilization of red mud waste for surface modification. The utilization of red mud for surface modification onto metal substrates had

been reported by Satapathy et. al. through plasma spray coating onto various metal substrates [10]. They used sieved red mud waste to obtain particle size of about 60-100 microns. The coating was performed by plasma spraying over aluminum, copper, mild steel, and stainless steel substrates with various applied torch input powers. By this method, the maximum hardness achieved was 560 HV.

Since there were almost no attempts on the utilization of red mud waste for surface modification, this research was aimed at utilizing red mud for surface modification. The coating was expected to improve the surface hardness and corrosion resistance of steel. In industrial processes, the hardness of steel is usually improved by alloying with increasing amount of noble metals, such as chromium, nickel, titanium, vanadium, etc. Alloying with such noble metals will indeed increase its price. Meanwhile, the corrosion protection in industrial processes was usually achieved by cathodic protection. However, this method is quite expensive although it offers good corrosion resistance. The coating by red mud through a chemical solution process, as is reported in this paper, can probably give an alternative for industrial scale applications by a relatively inexpensive and simple method. However, this method still needs further research and development to ascertain all the properties and behaviors of red mud-coated steel. In this research, the coating method was different from that in the work of Satapathy et. al. The coating of red mud was performed onto ST-37 steel through a dip-coating method. The coated steel was characterized using x-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), Vickers microhardness, and corrosion test by a weight loss method in 10% H₂SO₄ solution. The results showed improvements of corrosion protection and surface hardness of the coated steel. The maximum surface hardness of 1738.8 HV obtained was higher than that reported by Satapathy et. al. [10] although the method used was simpler.

Experiment

The red mud used in this research was obtained from the extraction of alumina from bauxite ore in West Kalimantan, Indonesia. Table 1 below shows the elements contained in the red mud are predominantly 32.77% Al₂O₃ and 37.58% Fe₂O₃ in mass percentage. A non-aqueous coating precursor was produced from a mixture of red mud powder, toluene as solvent, and polyethylene glycol 6000 (PEG 6000) as binder and plasticizer. Sesame oil was used as dispersant to prevent the agglomeration of red mud powder. The ratio of toluene and sesame oil as the liquid phase of the non-aqueous coating precursor was set at 59.1:40.9 %v/v, and the ratio of red mud powder and binder & plasticizer as solid phase was set at 99.97:0.03 at %wt/wt. Both phases, namely the liquid phase and the solid phase, were mixed with composition of 7 ml of liquid and 3 g of solid. Mixing was carried out by means of an ultra-turrax homogenizer at 20000 rpm for 10 minutes for each mixing step.

Table 1. Elemental composition of red mud by X-ray Fluorescence

No.	Elements	Content (%wt/wt)	No.	Elements	Content (%wt/wt)
1	Al ₂ O ₃	32.77	6	Na ₂ O	3.03
2	Fe ₂ O ₃	37.58	7	MnO	0.16
3	H ₂ O	19.33	8	MgO	0.14
4	TiO ₂	3.43	9	P ₂ O ₅	0.09
5	SiO ₂	3.40	10	CaO	0.06

ST-37 steel was used as the substrate and it was polished using a 2000 grit sandpaper, cleaned ultrasonically with detergent, acetone, and methanol, each for 5 minutes and then dried at ambient temperature to remove fat from the substrate. For complete cleaning of the metal substrate, the substrate was immersed in 1M NaOH for 10 minutes and rinsed with distilled water. The substrate was then immersed in 1M HCl for 10 minutes to improve its surface roughness and further rinsed with distilled water before being dried at ambient temperature. The coating process was carried out by a dip-coating method with a withdrawal speed of 0.23 mm/sec. The coating process was performed only once. The calcination process at 900, 1100, and 1300°C for 2 hours was carried out in nitrogen atmosphere to improve the morphology, crystallinity and chemical properties of the coating layer. Characterization was done using XRD (Philips Analytical X-Ray, Cu K α , $\lambda=1.54060$ Å) to examine the phases in the coating, SEM (JEOL JSM 6360LA Analytical SEM) to examine morphology of the layer, and EDS (JEOL JSM 6360LA) to examine elemental percentage of the probed area. The surface hardness of the prepared sample was examined by means of a micro Vickers hardness tester (Shimadzu Corporate M-85055) with 0.5 kg_f load for 15 seconds. The corrosion test was carried out by a weight loss method using 10% H₂SO₄ as the corrosion medium for 1, 3, 5, and 7 days. The samples were then cleaned from the corrosion products using continuous flowing water, immersed in acetone, and then dried in ambient condition. The corrosion rate (*CR*) can be calculated using the following formula [11],

$$CR = \frac{K.W}{A.T.D} \quad (1)$$

where *K* is a constant (8.76×10^4 mm/year), *W* is the weight loss (g), *T* is the immersion time (hours), *A* is the area of the sample (cm²), and *D* is the density of the sample (g/cm³).

Result and Discussion

Figure 1. shows the XRD pattern of untreated red mud. From this XRD result, it can be seen that the red mud has gibbsite (α -Al(OH)₃) and goethite (α -FeOOH) crystalline phases. The gibbsite shows the highest intensity that indicates the gibbsite has the highest content in this mixture phases. The other compounds composed by elements other than Al and Fe in Table 1 such as TiO₂ and SiO₂ are not detected. It was probably caused by the amorphous phase or the small amount of those compounds. The crystalline phases of Al₂O₃ and Fe₂O₃ are also not detected. The XRD result of this red mud is different than that has been reported by Yalçın et. al. [12] and Castaldi et. al. [13]. The red mud used in this experiment was composed by crystalline phases of two hydroxide-based compounds. On the contrary, the red mud used by Yalçın et. al. and Castaldi et. al. had crystalline phases of oxide-based compounds, namely hematite (α -Fe₂O₃), iron titanium oxide (Fe₂TiO₅), sodalite (Na₈(Cl,OH)₂Al₆Si₆O₂₄) and cancrinite (Na₆Ca_{1.5}Al₆Si₆O₂₄(CO₃)_{1.6}). The crystalline phase compounds contained in the red mud depend on its mineral resources, mineral resource's geography and their environmental condition. That is why the red mud has different crystalline phase compounds and compositions depending on their origin location.

