



Erosion wear of vinylester matrix composites in aqueous and acidic environments at elevated temperatures



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ABSTRACT

Slurry erosion wear performance of glass fibre reinforced vinylester composite (FRP) has been studied using a pilot-scale erosion test apparatus. Tests were conducted at elevated temperatures in aqueous and acidic environments. When using fine quartz as an abrasive material, FRP showed higher mass losses in the aqueous environment than in the acidic conditions, especially at higher temperatures. In this case, the FRP degradation was governed by the penetration of the used medium into the FRP structure. According to the absorption studies, the weight gain of the laminate was more pronounced in the water immersion compared to the acidic solution, which can be a prediction of an increased degradation rate and explain the higher wear in the aqueous medium. When the abrasive material was changed from fine to coarse quartz, the removal of the shielding matrix phase was extensive and a direct route for the acidic solution to the fibres was created causing more severe damage. This was also shown in scanning electron microscopy (SEM) studies, where the samples tested in the acidic solution showed extensive fibre flattening along the erosion flux. By increasing the test temperature close to the boiling point of the medium, an increase in the FRP wear could be seen. The increase in the rotation speed, on the other hand, did not automatically mean higher mass losses. This shows that the wear environment in the present test device is highly complicated with several interrelated parameters affecting the results.

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1. Introduction

Glass fibre reinforced polymers (FRP) are commonly used construction materials in challenging environments, such as chemical plants or industrial hydrometallurgical processes. The processing conditions in mineral leaching, for example, can be aggressive: the construction materials are exposed to sulphuric acid solutions (generally 0.5–15% H₂SO₄) that contain various ions, such as chloride, at elevated temperatures (up to 95 °C) [1]. The materials are subjected not only to corrosive fluid but also to solid-particle erosion: an effective mixing of constituents, including hard mineral particles with varying chemical composition, hardness, and shape is required to ensure high process performance. In industrial use, the impeller blades in the hydrometallurgical reactors are subjected to highly erosive conditions and turbulent flow, which depend on the geometry of the used system [2–4]. The particle flow inside the reactor may be highly complicated and, therefore, the erosion performance of a material in a real industrial application may differ considerably from the performance observed in a certain laboratory-scale test

system. Consequently, a test apparatus, which mimics the actual industrial application as closely as possible, should be designed to address this problem.

The performance of the FRP materials has been widely studied in aqueous [5–9] and acidic [10–13] environments, but hardly any studies combine erosion wear measurements in corrosive environment at elevated temperatures. It is well established that a prolonged exposure in water or in the acidic medium can damage the FRP structure, either in the fibre, resin, or in the matrix–fibre interface. Water, for example, can cause both reversible and irreversible changes in the resin, including hydrolysis, plasticisation, swelling, and microcracking. Reversible changes can be recovered by drying but irreversible changes will alter the mechanical properties permanently [8]. Moisture can also travel along the fibre–matrix interface, which can lead to detrimental effects in the fibre–matrix bond and, therefore, in the mechanical properties of the composite [14,15]. The diffusion rate is direction dependent: the rate is higher parallel to the fibres than normal to them [16]. Moisture diffusion is also faster in a composite that has exhibited mechanical damage [17]. Similarly, the moisture uptake level and the chemical processes that lead to the degradation of the composite are more pronounced as the temperature is raised [14]. The uptake of moisture diffusion is generally measured by weight gain

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and Fick's law is generally accepted model for predicting the water absorption behaviour of composites. However, non-Fickian behaviour can be observed in many cases due to the complicity of the absorption behaviour [18]. FRP materials may exhibit, for example, uniform material loss, localised pitting, fatigue, erosion, and delamination due to the acidic environment [16] and these may occur also simultaneously. The constituent responsible for the composite failures in the acidic environment is the glass fibres rather than the resin, which generally operates as a shielding barrier for the fibres [13].

A slurry pot type device has been commonly used to evaluate the erosion wear performance of different materials [19–24]. Generally, the tests have been conducted with varying speeds, impingement angles, particle sizes and shapes and it has given valuable information to be used e.g. in the material selection. Joshi et al. [22] have, for example, used a self-made slurry erosion pot to evaluate the erosion performance of Al_2O_3 filled glass/epoxy composite with varying impact angles, rotation speeds, and slurry concentrations. The results showed that the slurry concentration has the most significant effect on the wear rates while the test speed showed less impact on the results. It should be noted that in this type of tester, a precise control of the slurry velocity and the particle impingement angle is challenging due to the complexity of the flow in the pot and, therefore, the flow fields should be examined with the aid of computational fluid dynamics (CFD) modelling. In this study, used erosion test apparatus was constructed to resemble an industrial leaching reactor as closely as possible. During the design, CFD model was used to estimate the flow patterns inside the reactor. However, in this study, the viewpoint was fully empirical and the main goal was to examine the erosion wear of FRP in precise conditions set by the industrial application, where the mixing efficiency of the constituents is a key factor for a successful and a cost-effective process. The study is a continuation of the research performed by Sarlin et al. [25] on the slurry erosion wear of vinylester matrix composites in water medium with varying rotation speeds, abrasive size, and slurry temperatures. These tests were conducted mainly with the coarse quartz. It was noted that the highly complex wear conditions with several interrelated parameters caused some unexpected erosion mass losses. The dominating wear mechanism was abrasive wear, although at low rotation speeds the wear mode was rather fatigue-like. At high rotation speeds the cavitation erosion mechanism became more dominant. The FRP structure was found to be prone to both erosive turbulent flow and cavitation. Unlike metals, FRP was also sensitive to the abrasive concentration of the slurry and even low-energy particles caused wear [25]. In this paper, the tests in aqueous environment were continued with fine quartz and the results were compared to those obtained using sulphuric acid solution as the slurry medium.

2. Experimental details

2.1. Materials and sample characterisation

Since the goal of this study was to mimic the actual wear environment, the raw materials in the tested FRP laminates were also chosen based on the materials that are used in the FRP structures in the industrial mineral leaching processes. The tested vinylester laminate was manufactured by hand lay-up using epoxy vinylester resin (Derakane Momentum 411-350 supplied by Ashland). Vinylester has good durability in acidic conditions, since it has hydrolysable ester groups only at the ends of the polymer chains [5]. It also has more compact network structure than e.g. polyester showing hydrolytic degradation rather in the sample surface than inside the polymer bulk [26]. The manufactured

laminate contained six layers of chopped C-glass strand mat with a nominal weight of 300 g/m^2 and one layer of C-glass mat (30 g/m^2) on both laminate surfaces (glass fibre diameter ca. $16 \mu\text{m}$). The structure of the laminate is similar to the corrosion-resistant layer typically used in the surfaces of FRP structures employed in the leaching reactors. The goal of this C-glass barrier layer is to isolate the load bearing structural layer from the process fluid. The laminates were post-cured for 4 h at $80 \text{ }^\circ\text{C}$. The density was measured with a Wallace electronic densimeter, glass transition temperature (T_g) with a differential scanning calorimeter (DSC, model Netzsch DSC 204) under a nitrogen flow (50 ml/min) at a temperature range of $25\text{--}200 \text{ }^\circ\text{C}$ according to standard ISO 11357-1:2009. The fibre fraction of the laminates was measured with a residual ash determination test, where a small sample (weight of approximately 1 g) was cut from the laminate and its temperature was raised gradually to $600 \text{ }^\circ\text{C}$ in an oven. After all the resin had burned away, the remaining ash was weighed and compared to the original weight.

The erosion test samples (size $35 \text{ mm} \times 35 \text{ mm}$) were water jet cut from the laminate and the cut edges of the samples were sealed with vinylester resin (Derakane 441 supplied by Ashland) in order to avoid excess water intake. Vinyl ester resin was chosen as a sealant material to minimise the parameters affecting the results. To reach a fixed reference state, the samples were placed in an oven (6 h at $80 \text{ }^\circ\text{C}$) and then weighed. After the test, the worn samples were cleaned with a soft cloth and an ultrasonic cleaner in a water bath (1 h) to remove any abrasive material attached to the sample surfaces and to minimise the amount of loose wear debris that would affect the weight loss measurements. Finally, the samples were dried in an oven (6 h at $80 \text{ }^\circ\text{C}$), weighed, and the mass loss values for each sample were calculated. The wear surfaces were characterised with a scanning electron microscope (SEM, model Zeiss ULTRaplus) using an acceleration voltage of 10 kV . Prior to the SEM studies, the specimens were coated with a thin gold layer to avoid charging. The surfaces of the selected specimens were also characterised with an optical 3D measurement apparatus (Alicona InfiniteFocus G5).

2.2. Slurry erosion wear test set-up

Erosion wear tests were done with an in-house constructed erosion test apparatus (Fig. 1) that resembles the reactors that are used in real-life mineral leaching processes. The slurry erosion test apparatus has been designed with the help of CFD modelling to match the real-life leaching reactor as closely as possible in order to get reliable and comparable test results and it has been previously described in detail [25,27]. The sample holder in this test apparatus resembles a typical agitator type that is used in mixing solids in leaching processes. Twelve samples were attached to the agitator blades fixed at 45° position: half of them to the pressure side and another half on the suction side. The CFD model demonstrated that the wear pattern on the pressure side is more or less uniform, while a large trailing vortex is generated behind a moving impeller blade causing turbulence, which can lead to extensive localised wear. This behaviour has been widely verified in the literature [28–31]. The test construction allows a simultaneous study of two different wear phenomena, which, adding to the possibility to change the tip speed, slurry temperature, and slurry properties (type of abrasive and used liquid as well as slurry concentration), make this type of apparatus a valuable tool for optimising the service life of materials in real-life processes.

In this study, slurry erosion wear of FRP materials was studied in two different environments: water and 5% sulphuric acid (H_2SO_4 , concentration measured in wt%) solution. H_2SO_4 solution contained ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$, 0.5 g/l), which is often present in leaching environments. In this study, the role of ferric

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