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Influence of Particle Loading on the Microstructure of Aluminum-Fe₃0₄, Agte₂ Composite

Dankulu, M. H^{*,1}, Musa M. ², Ibrahim M. T², Abdulwaris A. D². Yerima Y. A². ¹Department of Energy and Applied Chemistry, Usmanu Danfodiyo University Sokoto, Nigeria ²Department of Mechanical Engineering, Usmanu Danfodiyo University Sokoto, Nigeria *Corresponding Email: <u>murtalahassandankulu@gmail.com</u>

ABSTRACT

Aluminum metal matrix composites (AMMC) are becoming viable materials for many applications in renewable energy and fuel security, entertainment, communications, machine tools, transportation, medical and pharmaceutical industries, and household appliances. In this study, Aluminum was reinforced with the Fe₃O₄.AgTe₂ through the recrystallization process, hence, AMMC was successfully developed. The aim was to locally develop new material with improved mechanical, and chemical properties that could be used as an absorber for solar thermal applications. The conventional Aluminum and composite were analyzed for morphological and chemical properties. Based on the obtained results, the microstructural analysis of the composite demonstrated an appreciable distribution of the reinforcement materials within the Aluminum matrix. The development of new phases was also revealed which is belief to have contributed immensely toward enhancing the strength and corrosion resistance of the composite. The conventional Aluminum sample are more corrosive in acidic solution than AMMC because the grain size of the AMMC materials being homogeneous; nevertheless, the grain size varied throughout the heating process, resulting in dislocation defects. Recrystallization creates homogenous grains when base materials are regularly intermixed during heating. It was observed that after the corrosion test in an acidic solution AMMC shows a positive outcome toward corrosion resistance (lower corrosion rate) this could enhance the thermal conductivity of the AMMC. It is therefore recommended that the composite can be used as solar thermal absorbers.

Keywords: Microstructure, Solar thermal, Corrosion Characteristics, Thermal conductivity, recrystallization

INTRODUCTION

Materials and energy were used by societies to improve their standard of living. The manipulation and processing of materials can be seen as a factor responsible for the emergence of global economies. The focus on materials to improve the overall performance of renewable energy technologies has motivated thermal energy professionals to seek and improve materials for solar thermal applications. Aluminum matrix composites (AMCs) have pure aluminum or its alloy as the matrix and are increasingly used in industrial and domestic applications due to their remarkable mechanical, material, and corrosive properties (Mannir, 2014). This led to the development of AMCs with every possible aluminum alloy matrix, incorporating various reinforcing materials to achieve the specific desired properties. AMCs can be manufactured by numerous methods according to their end use (Mukhopadhyay, 2012).

A previous study by Robb, (2020) reported that Natural composites are integral part of human life throughout the evolution of society. These natural composite materials include; stone, bones, woods, and even the bodies of living beings themselves represent a type of natural composite. arious new materials have emerged over time and as a result of the immense increase in demands for new and advanced facilities. The prominent areas such as health, sports, manufacturing, etc. are the focus of materials scientists and engineers. These areas require materials that exhibit higher specific strength, hardness, wear resistance, and dimensional stability at elevated temperatures. Metal matrix composites (MMCs) are proven to be able to meet the above requirements (Haghshenas, 2016).

Composites are multi-phase materials composed of matrices and reinforcements developed to meet the ever-increasing demand for attractive engineering materials. In general, composite materials have excellent thermal properties and excellent mechanical properties, including higher strength, hardness, and better corrosion resistance. These beneficial properties have led to increased use of composite materials in industrial and domestic applications (Abdudeen *et al.*, 2019). Aluminum matrix composites (AMC) are the next-generation composites that can replace singlereinforcement composites and introduce new features to improve the performance of engineering materials (Manikandan and Arjunan, 2020). AMCs are an environmentally friendly technology that has made strides in the engineering industry due to its various benefits. A number of factors are closely examined when considering the selection of materials to be used to perform a specific technical function (Joseph and Babaremu, 2019; Ajibola *et al.*, 2015).

Magnetite (Fe₃O₄), is an iron oxide ore mineral and one of the most important ores of iron, the most abundant magnetic mineral on earth (Ashrafi *et al.*, 2021). Magnetite is believed to be a suitable filler because it is abundant, inexpensive, and generates high free energy when reacting with aluminum. This reaction develops wettability between aluminum and magnetite and provides additional energy for the rest of the process (Wang *et al.*, 2021). In fact, magnetite can have

different chemical compositions depending on formation temperature, mineralization type, and genetic processes involved in its formation. For example, magnetite crystallizing from silicate and sulfide melts at high temperatures is expected to be enriched in Ti, Si, Al, Ba, Ta, Sc, and Y (Canil *et al.*, 2016).

Liao et al., (2018) studied corrosion, seen as an unavoidable process that destroys materials and their properties. Corrosion can be viewed as the deterioration of a material and its properties due to an electrochemical reaction between the material and its environment. Corrosion properties are a key indicator to consider when applying composites as construction materials. Reinforcing particles can chemically, physically, or electrochemically interact with the matrix and accelerate the rate of corrosion (Roseline and Paramasivam, 2019). A previous study by Akpoborie et al., (2021) reported that Corrosion is an electrochemical reaction; It is a reaction that involves the movement of electrons from one place to another. However, electrochemical reactions include both oxidation and reduction reactions; Oxidation reactions increase the material's valence number by removing particles from the material and charging it positively. It has been found that the galvanic interactions between the matrix and the reinforcement can also increase the corrosion rate. In general, anodic elements are more electropositive, meaning they don't lose their electrons. Cathodic elements are electronegative; hence they want to gain electrons. Therefore, it is believed that when mild steel is plated with zinc to form galvanized steel, zinc is compared to iron, consequently zinc should provide protection, serving as a sacrificial anode for the iron when the plating is disrupted to reveal small anodic areas of iron to expose and expose Corrosion can occur quickly. However, zinc protects iron in mild steel from corrosion in two ways. When the iron is fully coated with zinc, it forms a barrier between the iron carbide alloy and the environment, protecting the iron alloy below. If the zinc layer is scratched and the iron is exposed, the zinc will continue to protect the iron alloy as it tends to corrode (Access Engineering Library, 2011). This paper focused on the microstructure and corrosion characteristics of Aluminium metal matrix

composite reinforced with Al-Fe₃O₄-(AuTe₂) it is aimed to improve the thermal properties of the developed AMMC used for solar thermal applications.

Aluminum metal matrix composites can be produced by infiltration, powder metallurgy, squeeze casting, semi-solid and stir casting (Kavimani *et al.*, 2019). Various types of investigations have been carried out on composite materials with two or more reinforcements, such as Fe₃O₄-SiC reinforced with aluminum matrix hybrid nanofiller composites fabricated through powder

metallurgy method focusing on the investigation of hardness, strength, wear, and thermal properties (Negin *et al.*, 2020). Boutouta and Yacine, (2020) highlighted that the Taffel extrapolations indicate a singularity, as shown by the sample containing 40% Fe₂O₃ which had the best electrochemical performance due to its lowest corrosion rate and the lowest corrosion rate. Negin *et al.* (2020) revealed that Fe₃O₄ reinforced composites have higher corrosion resistance, compared to the aluminum matrix. Note that the detected pitting corrosion in aluminum base metal was not discovered in the existence of Fe₃O₄ nanoparticles. This might be in consequence of the existence of Al₃Fe and Al₅Fe₂ intermetallic, which serve as cathodes with regard to the metal matrix and enhance pitting corrosion resistance. The developed pitting behavior is more essential in the presence of chloride ions, while the aluminum surface becomes very vulnerable to pitting corrosion.

A previous study by Eftekhari *et al.*, (2017) concluded that Al₂O₃ improves corrosion resistance as Al₂O₃ affects anodic and cathodic reactions. Negin *et al.*, (2020) synthesized a hybrid nanocomposite of Al/Fe₃O₄/SiC using the powder metallurgy technique for Fe₃O₄ with varied weight percentages of 10, 20, 30, and 40 wt. % and with a constant weight percentage of SiC (20 wt. %), the results of this study show a homogeneous dispersion of the additive nanoparticles (Fe₃O₄ and SiC) into the Al matrix, which also improves micro-hardness and wear resistance. The preferred sintered density and micro-hardness were obtained for Al/30 Fe₃O₄/20 SiC as 2.69 g/cm³ and 91 HV, respectively, and the friction coefficient for the applied load 10 N decreased from 0.601 to 0.412 for the sample of Al/30 Fe₃O₄/20 SiC. The results of the corrosion resistance for Al/30Fe₃O₄/20 SiC is about 99.83%, and the corrosion resistance for Al/30 Fe₃O₄ is about 88.07%.

MATERIAL AND METHODS

Materials

All chemicals and reagents used in this study were of analytical grade. Aluminum matrix were obtained from the Sokoto Energy Research Centre (SERC), Usmanu Danfodiyo University Sokoto (UDUS). The 5% of H₂SO₄ was prepared by adding 30 ml of sulphuric acid to about 800 ml of distilled water as reported elsewhere (Chaubey *et al.*, 2016).

Collection and Preparation of Materials

Magnetite (Fe_3O_4) and silver-telluride ($AgTe_2$) were purchased from the local market, and then taken to the central advance science laboratory complex, Usman Danfodiyo University Sokoto, Nigeria, for identification. The materials were rinsed in deionized water for the removal of corrosion dust, crushed, ground, and sieved to obtain small particle sizes. Magnetite, and silver-telluride at different weight percentages, the samples were mechanically milled for 30 minutes using a Ball mill (Bm500, Antopaar, Austria), at 20 Hz at room temperature to ensure that additional components are distributed evenly and dispersed into aluminum matrix. The samples were mixed and placed on the aluminum matrix and inserted inside the furnace for heating at 600°C for 1hr. The sample mixture was allowed to cool in the furnace chamber, then heating was continued for 1 hour at 780°C. Later, the sample was removed from the furnace. A small amount of each of the samples was prepared by polishing with file and taken for analysis purposes of the SEM and XRD before and after the corrosion test.

		COMPOSITIONS (wt %)		
S/N	SAMPLE	Al	(AgTe ₂)	Fe ₃ O ₄
1	J	85	10	5
2	Κ	75	15	10
3	L	65	20	15
4	М	55	25	20
5	Ν	45	30	25

Table 1: Composition of aluminum, Magnetite and Silver telluride used

Corrosion Analysis

The specimens were polished and cleaned with acetone before being immersed in distilled water and air-dried. The starting weight of each specimen was computed before the corrosion test. The samples were immersed in 5% H₂SO₄ solution in glass beakers. The sample was kept at 28°C for 10 hours. At the end of the corrosion test, the sample was taken from the immersion solution and dried completely (after 3 hours). The final weights of the test sample were obtained using a digital scale (Alfattani *et al.*, 2022). After the corrosion test, the rate of corrosion in an acidic medium was evaluated using the formula below

$$Corr = 87.6 x (\Delta W/\rho at) mm/yr$$
(1)

Where Corr is the corrosion rate in mm/yr (weight loss measurements), K = 87.6, ΔW is the weight loss in g (W_b - Wa), ρ represents the density values of the base materials (2.71 g/cm³), a is the area of the sample in cm², and t represents the samples test time in hours (Alfattani *et al.*, 2022).

	pH Value	Temperature (°C)	Test time (Hours)
5% H ₂ SO ₄	5.28	28	10

SEM and XRD Analysis

Scanning electron microscopy (SEM). Scanning electron microscopy (Phenom prox SEM, generation 5, Switzerland), using a 2 kV acceleration voltage, the particle morphologies and microstructure were revealed. The microstructural investigation focused on the surface of sample. The samples were then subjected to Scanning electron microscopy to examine their microstructures. Energy Dispersive Spectroscopy (EDS) was carried out on the surfaces of the samples to uncover the chemical composition present in the composite. XRD analysis. X-ray diffractometer, (Rigaky mini flex model: XRD 300/600 Texas, USA) was used to record the XRD pattern of Aluminum reinforced with Fe₃O₄ and (AgTe₂) for phase analysis with a scan range from 3 – 90 operating at 40 kV and 15 mA, with a scanning speed of 10° 2-theta (20) and step size of 0.01°. The specimens used for surface morphological examination and phase pattern were immersed in the 5% H₂SO₄ solutions for 10 days, it was then removed, rinsed quickly with running water dipped in acetone, and dried. Scanning electron microscopy (SEM). Scanning electron microscopy (Phenom prox SEM, generation 5, Switzerland), using a 2 kV acceleration voltage, at the central advance science laboratory complex, Usman Danfodiyo University Sokoto, Nigeria, was used for the analysis.

RESULTS AND DISCUSSION

Microstructure Examination Results

The SEM micrograph for Al-10Fe₃O₄ and Al-20Fe₃O₄-25(AgTe₂) composites are presented in Figure 1a,b, respectively. Figure 1a shows the homogenous distribution of Fe₃O₄ particles in the Al matrix. Figure 1b shows (AgTe₂) as a gray-color element and Fe₃O₄ particles are the white-color elements which is distributed quite uniformly in the Al-20Fe₃O₄-25(AgTe₂) sample.



Figure 1. SEM Micrograph for two compositions: (a) Al-10 Fe₃O₄ and (b) Al-20 Fe₃O₄-25(AgTe₂)

Good reinforcement requires a bound principle with particles and a matrix. Chemical bonding and interdiffusion is the diffusion of atoms between two metals, and van der Waals bonding refers to the components of the interfacial mechanisms that involve filler and matrix bonding and the reaction between the matrix and reinforcements in the composite. The appropriate reaction between matrix and reinforcements promotes the wettability and bonding between them. The extreme reaction between particles and matrix can have an undesirable impact on the mechanical and thermal properties of the composite, while a violent reaction can damage the reinforcements (Jawalkar et al., 2017). In a previous study by Negin et al., (2020) It has been reported that an ideal reaction is desired for composite fabrication. Magnetite is commonly found in self-sustaining thermite reactions. Ferreira et al., (2016) reported that Magnetite (Fe₃O₄) is a suitable filler due to its low cost, excellent magnetic properties, and higher free energy of thermit reaction with aluminum. This reaction can improve the wettability between magnetite and aluminum matrix by providing additional energy to the process. However, soft magnetic materials have a narrow hysteresis loop, low Hc value, and high magnetic permeability with a narrow hysteresis loop, and low losses can be used as cores in electronic devices such as power transformers in AC adapters. For the Al-Fe-AuTe₂ composition, at a temperature above 600 °C, Al₂Fe₃AgTe₂ is at a stable phase, according to a similar study by Raghavan, (2009). Figure 2 shows SEM micrographs of Al $15Fe_3O_4-20(AgTe_2)$, Al-20Fe₃O₄-25(AgTe₂), Al-25Fe₃O₄-30(AgTe₂) and Al-30Fe₃O₄-35(AgTe₂) composite with uniform distribution Fe₃O₄ powders.



Figure 2. SEM micrographs of Al-Fe₃O₄-(AgTe₂) composites in different composite (a) Al- $15Fe_3O_4-20(AgTe_2)$, (b) Al- $20Fe_3O_4-25(AgTe_2)$, (c) Al- $25Fe_3O_4-30(AgTe_2)$ and (d) Al- $30Fe_3O_4-35(AgTe_2)$.

According to a previous study by Ravikumar, (2017), the mechanical properties of the hybrid composite depend on the proportion of reinforcement materials and the microstructure. The addition of silver telluride reformed the microstructure of Al-Fe₃O₄ composites hence, improved mechanical properties.

The four EDS analysis Al-Fe₃O₄-(AgTe₂) surface sample shown in Figure 3 were based on Figure 2. EDS analysis of the samples in Figure 2a shows the result for aluminum with 87.18 wt%. In Figure 2b, an AuTe₂ particle (gray color) exists in the aluminum matrix, with confirmed peaks of Ag (11.42 wt%) and Fe (1.53 wt%). Based on the EDS analysis, it revealed that the intermetallic phase of Al₃Fe and interfaces occurred at selected in figure 2c and confirmed in figure 3c and with Al 8.99 wt% and Fe (1.62 wt%) detected, where confirmed to be in accordance with the XRD results.



Figure 3a: The EDS spectrum of Al-15Fe₃O₄-20(AgTe₂)

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	Element	Element	Element	Atomic	Weight
	Number	Symbol	Name	Conc.	Conc.
	13	AI	Aluminium	70.20	25.79
	79	Au	Gold	26.71	71.64
	30	Zn	Zinc	1.19	1.06
	27	Co	Cobalt	0.78	0.63
	25	Mn	Manganese	0.57	0.43
	29	cu	Copper	0.30	0.26
	28	Ni	Nickel	0.24	0.19
0 1 2 3 4 5 6 7 8 9	10 11	12 13	14 15 16	17 18	19

Figure 3b: The EDS spectrum of Al-20Fe₃O₄-25(AgTe₂)



Figure 3c: The EDS spectrum of Al-25Fe₃O₄-30(AgTe₂)



Figure 3d: The EDS spectrum of Al-30Fe₃O₄-35(AgTe₂)

Therefore, by comparing all the microstructural images of Al-Fe₃O₄ and Al-Fe₃O₄-(AgTe₂) composite in Figures 1, and 2, we conclude that the addition of silver-telluride altered the microstructure of the Al-Fe₃O₄-(AgTe₂) composite. Figure 1b, the reinforcement in Al matrix (Al-20 Fe₃O₄-25(AgTe₂) are more rectangular in shape, while, in Figure 3a,b,c, and d by adding silver-telluride, the microstructure has evolved from rectangular to a more spherical shape.

XRD Analysis Results

It can be understood from Figure 4 that, the Aluminum matrix has a face-centered cubic structure (FCC). After the addition of Fe₃O₄ and (AgTe₂) particles into Aluminum, two peaks appeared, which refer to iron oxide and silver telluride (Ag₂Te) with two crystal systems, Orthorhombic and Cubic, before heat treatment. Moreover, after heat treatment, two peaks disappeared which were assigned to Fe₃O₄ and silver. After heat treatment, the XRD identified Al-Fe intermetallic compounds Al₃Fe and Al₂O₃. Similar behavior was reported by Eftekhari *et al.*, (2017) concluded in their results that Al₂O₃ improves corrosion resistance because Al₂O₃ affects the anodic and cathodic reactions.



Figure 4: XRD analysis for (a) Aluminum, (b) Magnetite, and (c) Al-Fe₃O₄.AgTe₂ before heating (d) Al-Fe₃O₄.AgTe₂ after treatment

The first peak is related to Fe₃O₄ with a cubic crystallography system. After adding Fe₃O₄ and (AgTe₂) particles into aluminum, and heat treatment, four main peaks were revealed for aluminum, one main peak for Fe₃O₄. $2\theta = (38.4^{\circ}, 44.70^{\circ}, 65.0^{\circ}, 70.1^{\circ}, 78.0^{\circ})$ were associated with aluminum, and $2\theta = (32.9^{\circ}, 35.3^{\circ}, 63.6^{\circ})$ was assigned to the Fe₃O₄ cubic crystal system. Therefore, this indicated that the intensity of the aluminum has decreased due to the influence of grain-sized refinement, after the addition of Fe in Al (Alfattani *et al.*, 2022).

Corrosion Test Results

However, the H₂SO₄ Solution formed an ash colored layer on the immersed surfaces of the samples which provided good corrosion resistance. The results of the corrosion test are shown in terms of sample weight before and after corrosion and weight loss due to the corrosion solutions, and the CR values for each sample are shown in Table 3.

Specimen	Solution	Weight of the	Weight of the	Weight Loss	Corrosion
		specimen	specimen	(g)	Rate (mm/yr)
		before corr	after corr test	ΔW	CR
_		test (g) Wb	(g) W _a		
AMMC	H_2SO_4	82	79.3	2.7	0.143736
Conventional	H_2SO_4	110	106	4	0.228040
Al					

Table 3: The Corrosion Test value of AMMC and conventional Al.

Alfattani *et al.*, (2022) reported that immersion corrosion test was considered more reliable and gave fairly accurate results compared to other corrosion tests. This form of testing makes it easy to identify and sort out materials that are unsuitable for a particular application. These tests are easy to perform for quick sample screening. Therefore, it is the most efficient and cost-effective way to evaluate material selection.

In this study, the corrosion and its impact on the AMMCs and conventional Al were assessed. The materials were subjected to a basic laboratory acid immersion test. The variance of corrosion in the examined samples was evaluated in Figure 5 to compare both AMMCs and conventional Al before and after corrosion and weight loss on corroded surfaces. The results of the immersion corrosion test are given as specimen weights before and after immersion, weight loss caused by corrosion in sulfuric acid solutions, and corrosion rate (CR) values for each specimen (Table 3). The results of the corrosion test showed a decrease in corrosion rate in an acidic solution shown in Figure 5. Weight loss in the AMMC was minimal (2.7g) in an acidic solution (H₂SO₄) and conventional Al was 4.0g in acid condition (H₂SO₄).



Figure 5: The variation of corrosion rate (CR) in an acidic solution (5% wt. H₂SO₄)

Weight loss sustained by the two samples after completing the corrosion test in the sulphuric acid solution for 10 h is presented in Figure 5. The conventional Al in an acidic solution had a higher CR (high weight loss) than the AMMC in an acidic solution. The CR of the AMMC was lower than the rates of the conventional Al, with the highest value being found in conventional Al.

However, corrosion properties are essential factors for consideration in applying composites as structural materials. The study by Roseline and Paramasivam, (2019) reported the reinforcing particulates might interact chemically, physically, or electrochemically with the matrix and accelerate the corrosion rate therefore, galvanic interactions between the matrix and reinforcement can also increase the corrosion rate.

However, the conventional Aluminum samples are more corrosive in acidic solution than AMMC because the grain size of the AMMC materials being homogeneous; nevertheless, the grain size varied throughout the heating process, resulting in dislocation defects. Therefore, recrystallization creates homogenous equiaxed grains when base materials are regularly intermixed during heating. The recrystallization of grains leads to improved mechanical and thermal characteristics.

CONCLUSIONS

- 1. AMMC were successfully developed to improve thermal conductivity properties, aluminum reinforced with magnetite-silver-telluride ore through a recrystallization method.
- 2. The microstructural analysis of the composite demonstrated the appreciable distribution of the reinforcement materials within the Al matrix.
- 3. The development of new phases was also revealed which therefore contributed immensely toward enhancing the strength and corrosion resistance of the composite.
- 4. By comparing the two samples, AMMC and conventional It is therefore recommended that Fe₃O₄-(AgTe₂) can be selected as a novel composite for solar thermal absorbers.

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