九州大学学術情報リポジトリ Kyushu University Institutional Repository

Extraction of Chromium Oxide from CCLW to Develop the Aluminium Based Composite by FSP as Reinforcement alongwith Alumina

Islam, Anas
Department of Mechanical Engineering, GLA University

Shashi Prakash Dwivedi G.L. Bajaj Institute of Technology & Management

Vijay Kumar Dwivedi Department of Mechanical Engineering, GLA University

Yadav, Rajat Department of Mechanical Engineering, GLA University

https://doi.org/10.5109/6625714

出版情報: Evergreen. 9 (4), pp. 993-1002, 2022-12. 九州大学グリーンテクノロジー研究教育センター

バージョン:

権利関係: Creative Commons Attribution-NonCommercial 4.0 International



Extraction of Chromium Oxide from CCLW to Develop the Aluminium Based Composite by FSP as Reinforcement alongwith Alumina

Anas Islam²*, Shashi Prakash Dwivedi¹, Vijay Kumar Dwivedi², Rajat Yadav²

¹G.L. Bajaj Institute of Technology & Management, Greater Noida, Gautam Buddha Nagar, U.P. 201310,

India

²Department of Mechanical Engineering, GLA University, Mathura, U.P. 281406, India

* Email ID: anasmechengg@gmail.com

(Received August 3, 2022; Revised November 18, 2022; accepted December 25, 2022).

Abstract: Chrome containing leather waste (CCLW) sometimes causes great harm to the environment as waste. However, the use of this waste can prevent environmental pollution. CCLW has been used in this investigation to extract collagen powder in the form of chromium. Particles made of ball-milled collagen powder were obtained after 100 hours of milling. Composite materials were created using the friction stir process (FSP). The microstructure of the composite produced in a single pass was found to be evenly distributed. Single tool pass composite generated by FSP demonstrated superior results in terms of hardness and tensile strength than composite developed in double and triple tool passes. With compared to the base aluminium alloy, tensile strength and hardness of composite material have increased by about 26.49% and 22.10% respectively. Composites made in a single tool run were shown to be more resistant to corrosion and thermal conductivity.

Keywords: Ball-milling; FSP; Cr₂O₃; CCLW; Leather waste

1. Introduction

Utilizing waste from industries is a very difficult task. Many times, these wastes rot due to non-utilization by the industries at the right time. Because of which these wastes produce many types of pollutions¹⁾. In today's time, mainly two types of pollutions such as soil and air pollutions are being generated more by industries wastes. Air pollution generated by wastes is very dangerous for human health. Several types of bacteria and viruses are born when waste material rots outside. These viruses enter the body with air through the mouth and nose of humans²⁾. This virus reaches inside the body of a person and causes various diseases. Many times, people die because of these diseases. However, if these wastes are used at the right time, the pollutions spread by them can be prevented³⁾.

Nowadays, various types of wastes are being generated from industries, which are very harmful to health. CCLW is also very harmful to human health. It is an extruded material treated as waste from the leather industries. If CCLWis not used again at the right time or it is not thrown in the right place, it can be very dangerous for human health. In the end, CCLW

decomposes mostly into air pollution, which causes significant problems for residents. ⁴⁾. However, CCLW has some elements which can increase the properties of other materials. Chromium oxide (Cr₂O₃) is the main component in CCLW. If Cr₂O₃ is extracted from CCLW properly, then this material can help in creating many new products. The presence of chromium (Cr) in chromium oxide always adds to the corrosion resistance of that material in which it is added and at the same time improves the mechanical properties of the base material⁵⁾.

Aluminum alloys' high corrosion resistance is another key attribute that makes them useful in engineering applications. However, many times it has been observed that some aluminium alloys lose their usefulness in many industries due to their low strength and hardness. Aluminium is widely used in the packaging industry to produce foam, cans, coils, and other wrapping materials. It is also used in making utensils and watch⁶. Aluminium is also used a lot in the construction industry. In the construction industry, it is used in the manufacture of wire, doors, window and roof. Aluminium and aluminium alloys are used in the transportation industry to produce aircraft, car bodies, spacecraft, bicycles, and marine parts. Today, many coins are made of alloy;

aluminium is also used as alloying elements with coins⁷).

Chromium is added to aluminium to refine the grain structure of aluminium. Addition of Cr in aluminium alloys improves the hardness as well as reduces stress corrosion sensitivity. Hence, after adding Cr to aluminium, there is a good increase in various properties of aluminium such as hardness, tensile and toughness^{8) 9)}. In this study, an attempt has been made to increase the properties of aluminium by adding Cr after proper extracting it from CCLW, as well as to prevent pollution caused by CCLW.

Aluminum-based composites are typically made using the casting method. However, numerous toxic gases are generated during the creation of composites using these methods, which is particularly detrimental to human health. These harmful gases are generated during the melting of the material. In today's time, the whole world is paying more attention to green manufacturing. The product is fabricated by green manufacturing approach without harming the environment. Due to these reasons, FSP is being used to develop composite. FSP is a green manufacturing process in which welding or composite of materials is carried out without generating any kind of harmful gases. While developing the composite by FSP, reinforcement is added to the materials without melting them¹⁰⁾. Table 1 shows the aluminium based surface composite developed by FSP with different reinforcement particles.

The FSP process is suitable in the fabrication of composite by using most of the ceramic and other particles including Al₂O₃, B₄C, SiC, graphene, graphite, Ni, RHA, TiC, TiN, Pine Needle Ash, CeO₂, CNTs, MoS₂, Al₃Ti, TiO₂ etc. Studies on the FSP of aluminium alloy and an Al₂O₃/collagen powder combination are very limited. The fundamental goal of this effort is to create a composite material out of aluminium and collagen powder (in the form of Cr) with alumina as reinforcement. Composites have been developed using the FSP process. The tensile strength, hardness, and roughness of the material were all measured. A corrosion test and a thermal expansion test are also included in this investigation to determine the influence of collagen and alumina addition in aluminium on corrosion and thermal behaviour of the created composite. The aluminium alloy AA6101 is used as the matrix material in this investigation, it contains 98.9% aluminium, 0.60 % magnesium, and 0.5 % silicon.

Table 1: Aluminium based surface composite developed by FSP with different reinforcement particles

S.No	Ref No.	Base Metal	Reinforcemen t	Remarks and conclusions
1	11)	Al	AlSi10Mg	Probability of porosity occurring reduced

2	12)	AA6061	SiC	Tilt
				angle(degree)
				= 2.5,
				Rotational
				speed (rpm):
				1400,
				Transverse
				speed
				(mm/min)=
				40, Number of
				passes =2
3	13)	AA2024	Al ₂ O ₃	Improved
		111202	111203	interparticle
_	14)	1 1 60 61	gig/41 o	bonding
4	14)	AA6061	SiC/Al ₂ O ₃	Wear
				resistance
				improved
5	15)	Al1050-H2	GNSs	The friction
		4		coefficient
				was reduced
				by Graphene
				particles
	16)	A 11050 TTO	E /E O	-
6	10)	Al1050-H2	Fe/Fe ₃ O ₄	AA1050/Fe
		4		and
				AA1050/Fe ₃ O
				4 based
				composite has
				been
				developed
	17)	1076	Al ₃ Ti	
7	1/)	A356		
7	17)	A356	A13 11	Strength and
7	17)	A356	Al311	ductility
7	17)	A356	Al311	ductility improved
7	17)	A356	Ai311	ductility improved after the
7	ŕ			ductility improved after the multi-pass
8	18)	A356	$B_4C + MoS_2$	ductility improved after the
	ŕ			ductility improved after the multi-pass
	ŕ			ductility improved after the multi-pass Improvement
	ŕ			ductility improved after the multi-pass Improvement in hardness and wear
8	ŕ	A356	B ₄ C + MoS ₂	ductility improved after the multi-pass Improvement in hardness and wear resistance
8	18)	A356 Al-Mg	$B_4C + MoS_2$ TiO_2	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined
8	18)	A356	B ₄ C + MoS ₂	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile
8	18)	A356 Al-Mg	$B_4C + MoS_2$ TiO_2	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved
8	18)	A356 Al-Mg	$B_4C + MoS_2$ TiO_2	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild
8 9 10	18) 19) 20)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel
8 9 10	18) 19) 20)	A356 Al-Mg AA5083	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear
8 9 10	18) 19) 20)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear resistance
8 9 10 11	18) 19) 20) 21)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C Gr/MoS_2	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear resistance improved
8 9 10	18) 19) 20)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear resistance
8 9 10 11	18) 19) 20) 21)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C Gr/MoS_2	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear resistance improved
8 9 10 11	18) 19) 20) 21)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C Gr/MoS_2 $Pine Needle$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear resistance improved Mechanical properties
8 9 10 11	18) 19) 20) 21)	A356 Al-Mg AA5083 Ti-6Al-4V	$B_4C + MoS_2$ TiO_2 $CeO_2 + CNTs$ B_4C Gr/MoS_2 $Pine Needle$	ductility improved after the multi-pass Improvement in hardness and wear resistance Grain refined Tensile strength improved Wear resistance of developed composite was better than stainless steel and mild steel Wear resistance improved Mechanical

wear

				characteristics improved
15	25)	Al	SiC/Al ₂ O ₃	Wear characteristics improved
16	26)	Al	TiC	Mechanical and wear characteristics improved
17	27)	Al	RHA	Distribution of reinforcement has been optimized optimized
18	28)	Al	Ni	Creep behaviour of the developed composite was observed

2. Materials and Methods

2.1 Collagen Powder Extraction

Collagen powder can be extracted chrome-containing leather waste by first soaking the leather waste in a solution of warm water and a mild detergent. This helps to break down the proteins and fats that make up the leather. The leather can then be processed in an acid bath to break down the collagen proteins even further. After the collagen proteins have been broken down, they can be extracted from the bath by filtering. The collagen powder can then be dried and used for various applications. The first step in obtaining collagen powder from CCLW was cleaning the leather waste. To dechrome it, sulphuric acid 37% (w/w) was used. Following dethroning, the dechromed solution was kept in a combination of HCl ethanol/EDTA (Ethylenediaminetetraacetic acid) 37% (w/w) for three days. To wash the dechromed CCLW, concentrated H2SO4 was used. For one day, the dechromed leather waste was soaked in a mixture of 0.1 M HCl ethanol, 0.2 M b-mercaptoethanol, and 0.05 M EDTA, with a sample-to-solution ratio of 1:30 (w/v). Finally, it was left in a 0.05 M acetic acid solution for one day. For 24 hours, collagen fibrils were allowed to settle. The collagen powder was then obtained by ball milling the material. Once more, alumina particles were added to the ball-milled collagen powder³³⁾. Additionally, collagen fibrils were ground into a powder using a ball mill. extracted Collagen powder is typically chrome-containing leather waste by a process known as acid hydrolysis. This process involves soaking the leather waste in an acid solution, such as sulfuric acid or hydrochloric acid, which breaks down the collagen fibers into smaller molecules. The solution is then filtered and heated to allow the collagen to separate from the other

components of the waste, before being collected and dried into a powder form. Step wise process of extracting collagen powder from CCLW is shown in Fig. 1. Ball-milling of collagen and alumina particles for roughly 100 hours is done to reduce the size of the particles, increase their surface area, and improve their dispersibility in a liquid. The increased surface area created by the ball-milling process allows for faster dissolution of the collagen and alumina particles in a liquid, which can be used to create a variety of products, such as medical implants, coatings, and adhesives. Additionally, ball-milling can be used to improve the mechanical properties of the particles, such as increasing their strength and improving their wear resistance. Fig. 2 shows the ball-milling of collagen and alumina particles for roughly 100 hours³²⁾. Powder XRD has been used to study the crystallinity and particle size of ball-milled collagen and alumina particles. XRD is a technique that was used to analyze the crystal structures of a material. It works by measuring the diffraction pattern of X-rays through the sample. This diffraction pattern has been used to identify the element and its crystalline properties, such as size, shape, and orientation of the crystalline domains. XRD was also be used to measure the particle size of ball-milled particles by analyzing the X-ray diffraction pattern and calculating the 2θ angle for each peak of the pattern. The 2θ angle is directly related to the size of the crystallites, with larger angles indicating larger particles. In addition, XRD can be used to study the structural changes in the ball-milled particles due to the mechanical processing, such as changes in crystallinity, crystallite size, and lattice parameters. XRD analysis of the powdered collagen and alumina particles is used to characterize the size, shape, and crystallinity of the particles. Also, XRD has been used to identify any impurities present in the particles. Additionally, XRD can be used to study the binding between collagen and alumina particles. XRD analysis of powdered collagen particles would reveal the three-dimensional structure of the collagen molecule. XRD analysis of alumina particles would reveal the crystalline structure of the alumina particles. The XRD spectrum would show the presence of distinct peaks corresponding to the crystalline structure of the alumina particles. XRD analysis of the powdered collagen and alumina particles is shown in Fig. 3. Powder XRD reveals Al₂O₃, Cr₂O₃, and Cr in the material.

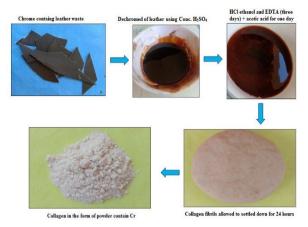


Fig. 1: Collagen powder extraction

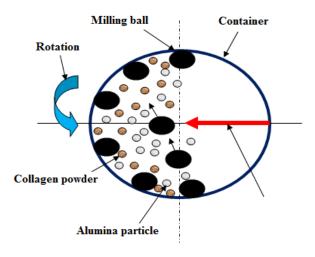


Fig. 2: Ball-milling of collagen and alumina particles

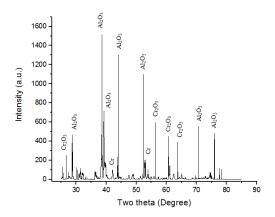


Fig. 3: Powder XRD of the ball-milled collagen and alumina particles

2.2 Experimental Procedure

Plates made of aluminum alloy, which are readily accessible commercially, were used as a matrix material. In this study, the number of tool passes on the workpiece surface was changed from 0, 1, 2, and 3. Collagen powder and Al₂O₃ particles of 30 to 40 nm in size were used to fill the groove²⁹). Friction Stir Process (FSP) is an

emerging manufacturing process which has been widely used to fabricate hybrid composite materials. The process consists of two stages; friction stir welding and friction stir forming. In this process, the material is heated and stirred by a rotating tool which creates a homogeneous and strong bond between the components. The process is advantageous due to the lack of any external heat source, making it a cost-effective and efficient way to produce hybrid composite materials. FSP is used to create hybrid composite materials by combining different materials such as metals, polymers, ceramics, and composites. These materials have unique properties which can be further enhanced by combining them together. For example, a hybrid composite material can be made by combining a metal and a ceramic, which can have better wear and corrosion resistance than a single material alone. The process of combining the materials is done by friction stir welding or friction stir forming, depending on the desired end product. FSP is widely used in the aerospace, automotive and marine industries to produce strong and lightweight components. In addition, it can also be used to produce components with complex shapes and geometries for various applications. The process can also be used to produce components with improved properties such as corrosion. Fig. 4 depicts the FSP procedure. The FSP parameters are shown in Table

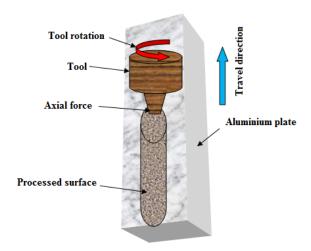


Fig. 4: Development of hybrid composite material using Friction Stir Process

Table 2: Process parameters

Sr. No.	Process Parameters	Values
1	Pin Length (mm)	3
2	Pin Diameter(mm)	6
3	Shoulder Diameter(mm)	18
4	Pin Profile of Tool	Threaded
5	Tool Tilt Angle (°)	0
6	Transverse Speed	25
	(mm/min)	
7	Rotational Speed (rpm)	1,000

The microstructure of the FSP samples was examined by polishing and etching them using Keller'sreagent (15ml HCL+25ml HNO3+ 10ml HF + 50ml H2O). The Tensometer (model-PC2200) has been used to perform the tensile test in the characterization section. Tensile specimen was prepared according to ASTM-E8-04 standards.

3. Results and Discussion

3.1 Microstructural Investigation

Fig. 5 shows the microstructure of FSP's surface composite. The microstructure of the FSP's of ball-milled collagen and aluminum surface composite shows a layer of collagen-aluminum composite material on the surface. The composite material consists of a homogenous mixture of collagen protein and aluminum particles. It has a microporous structure with small pores, which helps to increase the surface area. The average pore size is about 5 nanometers. The small pores help to increase the surface area and the ability of the material to absorb and release liquids. The increased surface area also helps to improve the mechanical properties of the material. Single tool pass composite microstructure is shown in Fig. 5 (a& b). Microstructure of the composite formed in the double pass and triple pass are shown in Fig. 5 (c) and Fig. 5 (d) accordingly. The microstructure of the FSP's of the ball-milled collagen and aluminum surface composite of the composite formed in the double pass and triple pass are quite different. In the double and triple pass, the FSP's are coarse, with large particles of collagen and aluminum being visible. In contrast, the FSP's of the single pass are much finer, with the particles being smaller and more uniform in size. This, in turn, resulted in a more homogeneous and finer microstructure. The single pass composite had a uniform microstructure, while the double and triple pass composites had a more heterogeneous microstructure with a higher amount of defects. The single pass composite had a higher hardness and tensile strength than the other two. The single pass composite also had superior fatigue resistance and impact strength compared to the double and triple pass composites. The single pass composite also had better interlaminar shear strength and mode I fracture toughness. Overall, the single pass FSP composite had superior properties and microstructure compared to the double and triple pass composites. The single pass composite had a more uniform microstructure with fewer defects, higher hardness, tensile strength, fatigue resistance, impact strength, interlaminar shear strength, and mode I fracture toughness. When examining the composite, it is clear that the ball-milled collagen and alumina particles have been well mixed with the matrix material throughout the fabrication process. This indicates that there is an increased degree of intermixing of the collagen and aluminum particles when the composite is not necessarily subjected to an additional

pass through the ball milling process. When the number of passes was increased from one to two and three, the agglomeration of reinforcement particles in the matrix material became more apparent. The mechanical characteristics of a composite will decrease if the reinforcing particles are agglomerated. Stir casting produces a variety of flaws, including porosity and shrinkage, in the composite material. After solidification, the microstructure of the stir-cast composite displayed a dendritic pattern. As a result, the mechanical properties of the composite are changed. It is clear from this study, microstructure of composites that casting problems such as porosity and shrinkage are completely eliminated when the composite is created using the FSP approach. As with FSW, the processing region of FSP is often separated into a base metal area, a stir zone (SZ), and a heat-affected area (HAZ). Thermal and mechanical processes during FSP affect the microstructure of the composite. Refined grains were found in the stir zone in this investigation to be uniform. These grains were decreased in size by a single run through a stirrer, as compared to the original metal. The foundation material with reinforcement was severely deformed by a high peak temperature created by the stir zone. This dynamic reintegration resulted in the formation of microstructures. A rise in high-angle grain boundaries following FSP resulted in a reduction in grain boundaries, which improved the flexibility of the base metal significantly. This resulted in grain formation when ball-milled collagen and alumina reinforcing balls were introduced to the aluminium alloy base metal. When fine particles were evenly dispersed during the recombination process, grain development was slowed. A pinning action at the grain boundaries caused this.

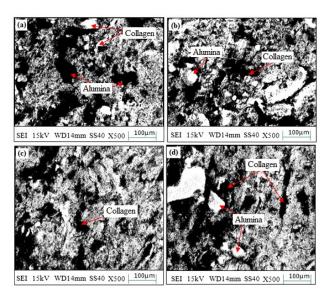


Fig. 5:(a & b) Composite developed in single tool pass, (c) Composite developed in doublé tool pass, (d) Composite developed in triple tool pass

3.2 Tensile Strength Analysis

Fig. 6 shows the composite's tensile strength after friction stir processing (a). More than double and triple passes, single tool passes resulted in an increase in the composite's tensile strength value. In comparison to the base material, the composite's tensile strength was improved by 26.89% after a single pass. The presence of Cr in collagen and the hardness of alumina particles are the primary causes of this increase in tensile strength. The grain structure was fine-tuned by FSP after the collagen and alumina combination was ball-milled and blended with aluminium. Additionally, the basic material's tensile strength was improved by Sample size introduction of hard phases. monocrystalline materials and average grain size in polycrystalline materials influence tensile strength in most metals. Plastic deformation begins during the friction stir processing growth of the composite. Both small grain structure and coarse grain structure are possible during plastic deformation. increases. Increasing the tensile strength of materials is constantly hindered by materials with coarse grain architectures.

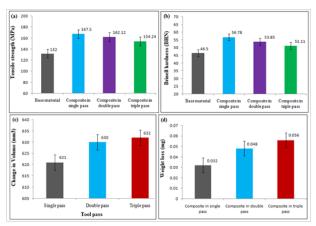


Fig. 6: (a) Tensile strength, (b) Hardness, (c) Thermal expansion behaviour, (d) Corrosion loss

After a very gradual and gentle plastic deformation via FSP, a very weak crystal dislocation may be created. When gradual dislocation occurs, the likelihood of finer grain structure is increased. Strengthening a material by reducing the grain size has long been a priority. As the dislocation rate rises, the likelihood of developing coarser grain structure.

3.3 Hardness Analysis

Compound reinforced with ball-milled collagen and Alumina particles was produced by FSP. Fig. 6 (b) illustrates the hardness of the composite material. The composite's hardness as a result of the single-pass process was clearly visible. The hardness of the single-pass composite was determined to be 56.78 BHN. By analyzing this data, it can be concluded that when the combination of ball-milled collagen and aluminium

particles was merged into aluminium, the hardness of the composite was improved by around 22.10%. During the creation of composites via FSP, it is critical to guarantee that the composite has a fine grain structure. Composites should be produced using the best FSP parameters in order to get the best results.

3.4 Thermal Expansion Behavior

When the material can be utilized at greater temperatures, its utility grows. Parts utilized in all of these sectors, from manufacturing to automobiles, operate best at greater temperatures. Because of this, a thermal expansion test must be performed on the composite material. The behaviour of composite material's thermal expansion was thus detected to examine whether or not it is suitable for use in a high-temperature environment. Before the thermal expansion test, the dimensions of all the samples were recorded. Before the test, all dimensions were the same. All of the composites were maintained to the same 625 mm3 size (25 x 5 x 5). For 48 hours, all of the composite samples were held at a constant temperature of 450°C in a muffle furnace. Six to seven thermal samples were obtained for each pass.

There was a discrepancy in the samples' dimensions after the thermal expansion test. At the same temperature, the sample with the least amplitude change performs better. Because of this, the same sample should be used in a high-temperature setting. Fig. 6 depicts the composite's thermal expansion across a range of passes (c). By increasing the number of passes of FSP in the formation of composites, the volume of the composite samples grew, according to the findings of thermal expansion. There was a minimum volume difference of 4mm3 and a maximum volume difference of 7mm3 detected in the formation of composites, respectively. The FSP approach for producing the composite is now more often employed thanthe stir casting process. " This is mostly due to the formation of the composite's grain structure and grain size during solidification. Composite produced by FSP has a superior grain structure than composite produced by stir casting, according to this research. In the FSP method, the composite is created in a solid state. When using the FSP approach, the material's grain structure is more stable. The mechanical properties of the composite as well as the thermal expansion properties increased dramatically due to the systematic grain structure. Here, single-pass composites have superior thermal properties than double- and triple-pass composites. Single-pass composite had a fine grain size and appropriate grain structure³⁰⁾. Because the grain size was already finer, it was less likely to shrink during the thermal expansion test. The three passes resulted in a coarser and more unstable grain structure in the composite. When the composite was held at a higher temperature, it shrank more because of the coarse grain's

size31).

3.5 Corrosion Behavior

It is very necessary to find the corrosion behaviour of any material when the demand for using that material is in a high moisture environment. In such a situation many times a material with a very good mechanical property also gets rusted. Surface qualities like hardness and wear resistance are deteriorating because of this. As a consequence, the substance is rendered useless. As a result, investigating the new material's corrosion behaviour becomes essential. The FSP composite was monitored for 120 hours in 3.5 weight percent NaCl in this investigation. Samples were weighed before and after the test. The composite's minimal weight loss indicates its efficacy. When the composite was created in a single pass, the least amount of weight was lost. Fig. 6 shows that the single-pass composite generated by this method had a corrosion loss of 0.032 mg (d). Because of the collagen's inclusion of Cr2O3(~25%-30%), the resulting composite has improved corrosion resistance. The FSP did, however, improve the composite's grain structure in a single pass. The composite's corrosion weight loss was minimized as a result of the improved grain structure.

3.6 XRD Analysis

In order to determine the molecular and atomic structure of a crystal, X-ray crystallography (XRC) is employed. In this procedure, an incoming beam of X-rays is diffracted in many different directions due to the crystalline structure.

Scientists that study crystals create a three-dimensional image of the electron density within the crystal by analyzing the angles and intensities of diffracted beams. The average atomic locations in the crystal, determined by its electron density. Traditional X-ray diffractometers have the X-ray detector, the sample holder, and the X-ray tube.

In a cathode ray tube, X-rays are created when electrons are produced by heating a filament, accelerated towards a target by supplying a voltage, and then bombard the target material. Characteristic X-ray spectra are generated when the electrons have enough energy to eject electrons from the inner shells of the target material. The wavelengths used are particular to the item being studied (Cu, Fe, Mo, Cr).

The material is illuminated by a focused beam of X-rays. While the sample and detector rotate, an intensity reading of the X-rays which are reflected is taken. The Bragg peak appears when the X-rays incident on the sample satisfy the Bragg Equation. This X-ray radiation is picked up by a detector, processed, and transformed into a count rate before being sent to an output device, such as a computer monitor or a printer. The sample in an X-ray diffractometer revolves at an angle in

comparison to the collimated X-ray beam and the X-ray detector, which accumulates the diffracted X-rays, rotates at an angle of two relative to the sample. A goniometer is the tool used to hold the angle and spin the sample. Exhibited in Fig 7 is the XRD behaviour of the composite generated here using the optimal combination of FSP settings. The generated composite material's XRD was studied to determine its constituent phases. The X-ray powder diffraction analysis confirmed the presence of aluminium, aluminium oxide, chromium, and chromium oxide. The improved tensile strength and hardness of the produced composite may be traced back to the incorporation of hard phases like Al₂O₃, Cr, and Cr₂O₃. The XRD data shows that the formation of Al2Cu in the composite was successful. This indicates that the FSP process achieved the desired result of combining different materials to produce a new alloy. This newly formed alloy is responsible for the improved mechanical properties of the composite. The XRD results show that the composite is composed of Al, Al2O3, Cr2O3, and Al2Cu. This suggests that the FSP process was capable of producing a strong and durable composite material, which could be used in a variety of applications.

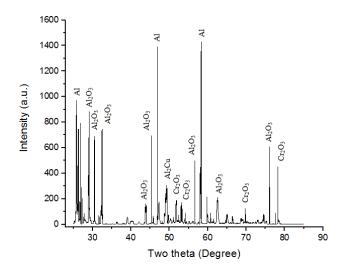


Fig. 7: XRD Analysis

After Al, Al₂O₃, Cr₂O₃, and Al₂Cu in the manufactured composite, the XRD results indicate the greatest peak to be Al. The XRD pattern also indicates that there is a significant proportion of Al₂Cu present in the manufactured composite. This indicates that the FSP process has been successful in forming a compound from the aluminum and copper phases. The presence of Al₂Cu suggests that the composite has a higher degree of homogeneity, which can result in improved mechanical properties. The XRD results also suggest that the composite contains small amounts of other phases, such as Cr₂O₃ and Al₂O₃, which can contribute to the mechanical properties of the composite.

4. Conclusions

The presence of Cr in CCLW proved to be good reinforcement particles. Collagen powder in form of Cr₂O₃ was extracted from CCLW and ball-milled with alumina particles. After ball milling, we were able to get reinforcement particles as a single unit. The surface composite was developed using the friction stir process (FSP) technology. Using a microstructure picture, it was shown that a combination of ball-milled collagen and aluminium particles reinforced by FSP may be created. The distribution of reinforcement in a single tool pass was found to be satisfactory. Composite materials created with just one tool pass had the highest tensile strength and hardness scores, too. The distribution of reinforcement in multiple tool passes was found to be more varied. The tensile strength and hardness scores for composite materials created with multiple passes were lower than those created with a single pass. However, multiple passes did increase the surface finish of the composite materials, allowing for additional customization. In comparison to the base aluminium alloy, the tensile strength and hardness improved by 26.89 percent and 22.10 percent, respectively. These additions increased the alloy's strength by forming strong intermetallic compounds and a harder oxide layer on the surface of the alloy. The improved mechanical characteristics of the composite were attributed, according to the XRD data, to newly formed phases within the composite, such as Cr₂O₃ and Al₂O₃. The composite produced by a single tool pass exhibits the least weight loss and expansion due to corrosion and corrosion-induced thermal expansion. 0.032 mg of corrosion weight loss and 4 mm³ of thermal expansion were discovered in the composite, which is adequate. The composite produced by the single tool pass is suitable for the intended applications and can be used for various applications without any further processing.

Nomenclature

CCLW	Chrome containing leather waste
FSP	Friction Stir Process
Cr ₂ O ₃	Chromium oxide
Cr	Chromium
Al ₂ O ₃	Aluminum oxide
B ₄ C	Boron carbide
SiC	Silicon carbide
Ni	Nickel
RHA	Rice husk ash
TiC	Titanium carbide

TiN	Titanium nitride
CeO ₂	Cerium dioxide
CNTs	Carbon nano tubes
MoS ₂	Molybdenum Sulphide
Al ₃ Ti	Titanium aluminide
TiO ₂	Titanium dioxide
Al ₂ Cu	Aluminiumcopper (2/3)
Cu	Copper
Fe	Iron
Мо	Molybdenum
Cr	Chromium
w/v	Weight/volume

References

- F. Taufany, M.J. Pasaribu, B.Y.S. Romaji, Y. Rahmawati, A. Altway, S.Susianto, Nurkhamidah, J.G.A. Mursidah, F. Desi, S. Yulianti, D. Rahmawati, D. and G. Stellarosari, "The Synthesis of Activated Carbon from Waste Tyre as Fuel Cell Catalyst Support," Evergreen., 9(2) 412-420 (2022).
- S.P. Dwivedi, M. Maurya, and S.S Chauhan, "Mechanical, Physical and Thermal Behaviour of SiC and MgO Reinforced Aluminium Based Composite Material," Evergreen., 8(2) 318-327 (2021).
- 3) A. Mandal, M.K. Farhan, and T.P. Sastry, "Effect of reinforced Al2O3 nanoparticles on collagen nanobiocomposite from chrome-containing leather waste for biomedical applications," Clean Techn Environ., **18** (3), 765–773 (2016).
- 4) S.P. Dwivedi, "Effect of ball-milled MgO and Si3N4 addition on the physical, mechanical and thermal behaviour of aluminium based composite developed by hybrid casting technique," International Journal of Cast Metals Research., 33 (3), 35-49 (2020).
- 5) S. Ray, "Effect of Control Parameters on Erosion Wear Performance of Glass-Epoxy Composites Filled with Waste Marble Powder," Evergreen., 9(1)23-31 (2022).
- 6) S.P. Dwivedi, and V.R. Mishra, "Physico-Chemical, Mechanical and Thermal Behaviour of Agro-waste RHA-Reinforced Green Emerging Composite Material," Arabian Journal for science and engineering.,44 (2), 8129–8142 (2019).
- S.P. Dwivedi, and A. Saxena, "Extraction of collagen powder from chrome containing leather waste and its composites with alumina employing different casting techniques," Materials Chemistry and Physics.,253 (3), 123274 (2020).

- 8) C.V. Singh, P. Pachauri, S.P. Dwivedi, S. Sharma, and R.M. Singari, "Formation of Functionally Graded Hybrid Composite Materials with Al2O3 and RHA Reinforcements using Friction Stir Process," Australian Journal of Mechanical Engineering., 20 (1) 141-154(2019). https://doi.org/10.1080/14484846.2019.1679583.
- 9) A.H. Maamoun, S.C. Veldhuis, and M.Elbestawi, "Friction stir processing of AlSi10Mg parts produced by selective laser melting," J. Mater. Process. Technol., 263 (2)308–320 (2019).
- 10) P.B. Prangnell, and C.P.Heason, "Grain structure formation during friction stir welding observed by the stop action technique," Acta Mater., **53**(3) 3179–3192 (2005).
- 11) K. Yang, W.Y. Li, P.L.Niu, X.W. Yang, and Y.X. Xu, "Cold sprayed AA2024/Al2O3 metal matrix composites improved by friction stir processing: Microstructure characterization, mechanical performance and strengthening mechanisms," J. Alloy. Compd., 736(3) (7) 115–123 (2018).
- 12) N. Parumandla, and K.Adepu, "Effect of Al₂O₃ and SiCnano reinforcements on microstructure, mechanical and wear properties of surface nanocomposites fabricated by friction stir processing," Mater. Sci. Medzg., **24**(2) 338–344 (2018).
- 13) A. Jain, C.S. Kumar and Y. Shrivastava, "Fabrication and Machining of Metal Matrix Composite Using Electric Discharge Machining: A Short Review," Evergreen., **8**(4)740-749 (2021)
- 14) J.B. Fernandez, E.J. Macias, J.Muro, L.S.Caputi, D. Miriello, R. De Luca, A.S. Roca, and H.D.C. Fals, "Tribological behavior of AA1050H24-graphene nanocomposite obtained by friction stir processing," Metals.,8(1), 113 (2018).
- 15) E.R.I Mahmoud and M.M. Tash, "Characterization of aluminum-based-surface matrix composites with iron and iron oxide fabricated by friction stir processing," Materials., 9 (1) 505 (2016).
- 16) R. Yang, Z.Y. Zhang, Y.T. Zhao, and G. Chen, Y.H. Guo, M.P. Liu, and J. Zhang, "Effect of multi-pass friction stir processing on microstructure and mechanical properties of Al3Ti/A356 composites," Mater. Charact., 106 (3), 62–69 (2015).
- 17) R. Srinivasu, A.S. Rao, G.M. Reddy, and K.S. Rao, "Friction stir surfacing of cast A356 aluminium-silicon alloy with boron carbide and molybdenum disulphide powders," Def. Technol.,11 (1) 140–146 (2018).
- 18) S.A Hosseini, K. Ranjbar, R.Dehmolaei, and A.R.Amirani, "Fabrication of Al5083 surface composites reinforced by CNTs and cerium oxide nano particles via friction stir processing," J. Alloy. Compd.,622 (5) 725–733 (2015).
- 19) G.M. Reddy, A.S. Rao, and K.S. Rao, "Friction stir surfacing route: effective strategy for the

- enhancement of wear resistance of titanium alloy," Trans. Indian Inst. Met., **66** (2), 231–238 (2013).
- 20) M.K. Gupta, "Analysis of tribological behavior of Al/Gr/MoS2 surface composite fabricated by friction stir process," Carbon letters., **30**(4) 399-408 (2020).doi: 10.1007/s4282 3-019-00109 –w.
- 21) M.K. Gupta, P.K. Rakesh, and I. Singh, "Application of industrial waste in metal matrix composite," J Polym Compos., 4(3), 2321–8525 (2016).
- 22) G. Hussain, R. Hashemi, H. Hashemi, and K.A. Al-ghamdi, "An experimental study on multi-pass friction stir processing of Al/TiN composite: some microstructural, mechanical, and wear characteristics," Int J Adv Manuf Technol.,84 (5) 533–546 (2016).
- 23) Mahmoud, E.R.I., Takahashi, M., Shibayanagi, T., Ikeuchi, K. Wear characteristics of surface-hybrid-MMCs layer fabricated on aluminum plate by friction stir processing. Wear., **268**(9–10), 1111–1121 (2010).
- 24) S. Jerome, B.S. Govind, B.S.P.Kumaresh, and B. Ravisankar, "Influence of microstructure and experimental parameters on mechanical and wear properties of Al-TiC surface composite by FSP route," J Miner Mater Charact Eng., 11(5) 493–507 (2012).
- 25) A. Kumar, A.K. Chanda, and S. Angra, "Numerical Modelling of a Composite Sandwich Structure Having Non Metallic Honeycomb Core," Evergreen., 8(4)759-767 (2021).
- 26) Yadav, R., Islam, A. and Kumar Dwivedi, V. (2022), "Wear and microstructure analysis of aluminium based composite using eggshell and rice husk ash as reinforcement", World Journal of Engineering, https://doi.org/10.1108/WJE-05-2022-0186.
- 27) A. Islam, S.P. Dwivedi, and V.K. Dwivedi, "Effect of friction stir process parameters on tensile strength of eggshell and SiC-reinforced aluminium-based composite," World Journal of Engineering., (2020). doi:10.1108/WJE-08-2020-0387.
- 28) R. Yadav, V.K. Dwivedi, A. Islam, and S.P.Dwivedi, "Analysis of mechanical properties of Al-based metal matrix composite reinforced with ES and RHA," World Journal of Engineering., (2021).doi: 10.1108/WJE-02-2021-0079
- 29) R. Yadav, S.P. Dwivedi, and V.K. Dwivedi, "Synthesis and mechanical behavior of ball-milled agro-waste RHA and eggshell reinforced composite material," Materials Performance and Characterization., **10**(1), 237-254(2021).
- 30) A. Islam, S.P. Dwivedi, V.K. Dwivedi, S. Sharma, and D. Kozak, "Development of marble dust/waste PET based polymer composite material for environmental sustainability: fabrication and characterizations," J. Mater. Perform. Charact., 10 (5), 538-552(2021).

- 31) V.K. Dwivedi, S.P. Dwivedi, and R. Yadav, "Effect of heat treatment process on microstructure and mechanical behaviour of Al/MgO composite material," Adv in Mat and Pro Tech., 1-10 (2020).doi: 10.1080/2374068X.2020.1829955.
- 32) S. P.Dwivedi, A.Saxena, S.Sharma, G. Singh, J. Singh, M. Mia and S. Wojciechowski, "Effect of ball-milling process parameters on mechanical properties of Al/Al2O3/collagen powder composite using statistical approach". J of Mater. Res. and Tech, **15**(1), 2918-2932(2021).
- 33) M. Mehra, S. P. Dwivedi, N. Kumar, N. Thukral, and Y. Singh. "Extraction of Collagen from Leather Waste to Develop Aluminium Based Metal Matrix Composite." Materials Today: Proceedings, **25** (1), 581-585 (2019).