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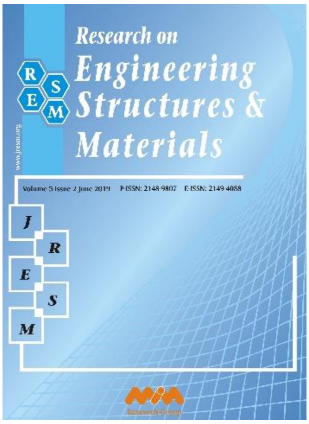

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Research Group

Research Article

Investigation of recycled aluminum matrix composites reinforced with copper and γ-alumina manufactured by sinter and sinter + forging methods

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Abstract

The present work reviews the toughening mechanisms and microstructural analyses of recycled hybrid metal matrix composites (aluminium based) reinforced with γ-alumina and pure recycled copper. This composite was manufactured by sintering and sinter + forging called the combined process. To analyze the mechanical / physical behavior of hybrid metal matrix composite materials; quasi-static compression, three-point bending (3PB), low velocity impact (dynamic compression) experiments were carried out. Additionally, wear and creep tests were conducted with a nanoindenter to evaluate the wear and time dependent behaviour of this composite. Evaluation of the microstructure of hybrid metal matrix composites were performed with Scanning Electron Microscope (SEM) supported by Energy Dispersive Spectrometer (EDS) analysis. The results showed that the composites have homogeneous structure without porosity and very homogeneous distribution of fine γ-alumina (Al_2O_3) and copper particles. Sinter + forging process yielded a material that had higher strength, hardness and better resistance to wear. This composite will be targeted for linkage applications where high toughness and high surface damage resistance is required.

1. Introduction

A new family of aluminium-based composites is extensively being used in the different industrial areas (aerospace, automotive etc.) doped with different metal and ceramic reinforcements to improve their mechanical and tribological properties [1-8]. When the γ-alumina ceramic reinforcement is added to the aluminum matrix, it can significantly increase the wear resistance along with the mechanical / physical properties of such hybrid metal matrix composite materials. The addition of copper reinforcement together with γ-alumina can give a good combination of high ductility and high strength [5, 9, 13-18].

The manufacturing operation that we determine the combined sinter+forging process used to manufacture the aluminium-based composites can offer simplicity in processing and lower the manufacturing costs as these composites are produced from recycled constituents known as fresh scrap materials. In this current research, novel composite reinforced with γ-alumina and copper was designed for possible use in linkage/connector parts in aeronautical applications [7, 11, 16].

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The composite design process from fresh scrap recycled components can be applied very well to optimally economical industrial parts for a minimized cost and fast production. As can be seen in the researches in the literature, the sinter+forging process is a manufacturing process that is defined as a process of forming close to high accuracy for the manufacturing of parts processed from fresh scrap materials [6-14]. Basically, this manufacturing method is the preferred method for bulk materials in different industrial areas. With a low-cost sintering accession for processing fine particle reinforced composites, it is ensured that industrial products always show a high performance under different service conditions. An economically minimized sintering approach for the manufacturing of fine particle reinforced composites provides high efficiency of mechanical behaviors of parts used in different industrial applications such as static compression, impact loading, 3-point bending, creep and fatigue. This principle set forth in our study can be applied to many other composite-based materials in many different industrial areas and at different scales. It reveals that very hard and strong parts can be attainable with this combined manufacturing method, but the fact that it can be performed at a lower cost than other processes suggested in the literature can be considered as one of the most important property of this method.

This material developed in this study based on the results of quasi-static compression, impact and wear tests can be recommended for use in areas where wear and high impact forces are expected.

2. Experimental Conditions

Within the scope of this study, an alternative hybrid metal matrix composite (aluminium based) with optimum economy was manufactured using fresh scrap recycled chips of two aluminum series Alumix-431 (50% wt) and AA1050 (50% wt) supplied by a French aerospace company. The two aluminum series (after atomization) were mixed by planetary ball mill for 1 hour by high energy milling and doped with copper γ - Alumina (Al_2O_3). The final composition was homogenized for 4 hours by ball milling. Pure nano-aluminum (<3-5 wt %) powder was added to the mixture to obtain a homogeneous mixture with good wettability of the reinforcements with the matrix. The final composition is given in Table 1 and the chemical composition of Alumix-431 supplied by the aerospace company in Table 2 (Analyzes were carried out by the relevant aerospace company).

Table 1. Compositions of the designed composite (wt. %)

Specimen Name	Alumix-431+AA1050 (Matrix, 1:1)	Cu	γ - Al_2O_3	Zn-St
MENG	Balance	25	15	2

Table 2. Scrap Alumix-431 (wt. %) chemical composition

Element	Al	Cu	Mg	Zn
wt. %	91.45	0.55	2.5	5.5

Microstructural analysis was carried out with SEM (JEOL USA, Inc., Peabody, MA, USA). The dispersion of reinforcement particles in the matrix and the interface in the matrix / reinforcements were also analysed. Microhardness tests ($HV_{0.1}$) were conducted on polished and etched specimens. The measured microhardness values are given in Table 3 with an accuracy of ± 15-20% for the two manufacturing operation techniques, alternately.

Table 3. Measurements of the microhardness values of the composites

Composition Name	Micro Hardness Values, $HV_{0,1}$ (at interface only)
MENG (Sintering)	245± 20
MENG (Sinter+Forging)	290± 15

The density measurements of composite specimens using Archimedes method were performed. These density values changeable between 2.95 and 3.45 g/cm^3 with ± 5% accuracy, respectively. Quasi-static compression tests (DIN 50106) were conducted using Zwick (Z250) mechanical test system at 1 mm/min strain rate. An average of 3-4 cylindrical specimens (Height/Diameter ≥ 1.5) were used for the test specimens produced by each manufacturing method. 3PB tests (ASTM D790) were also performed using the same mechanical test system. Low velocity impact (drop weight) tests were carried out with a drop tower (developed in SUPMECA/Paris Quartz Laboratories) to analyse the response of these composites designed with metal matrix to dynamic loading (Total weight 2.2 kg; Punch height 150 mm; Impact velocity~ 2.8 m/s).

Subsequently sintering and/or sinter + forging manufacturing processes, whole cylindrical specimens were tested for machinability at high cutting speed with low cutting force [6, 9, 16]. The purpose of this process is to remove damages on the surface of cylindrical specimens in cases such as work hardening or damage to the cutting tool.

Wear tests were performed by applying a normal load of 50 mN on a linear wear track of 500 μm and 1000 μm for 50 cycles and using the scratch ability of a nanoindenter. A cycle used during the tests was defined as the departure and the return on the same path. The wear tests were repeated a total of 10 times for each specimen, using a conical tip with a cone angle of 90 ° and at a speed of 50 μm/s (tip speed).

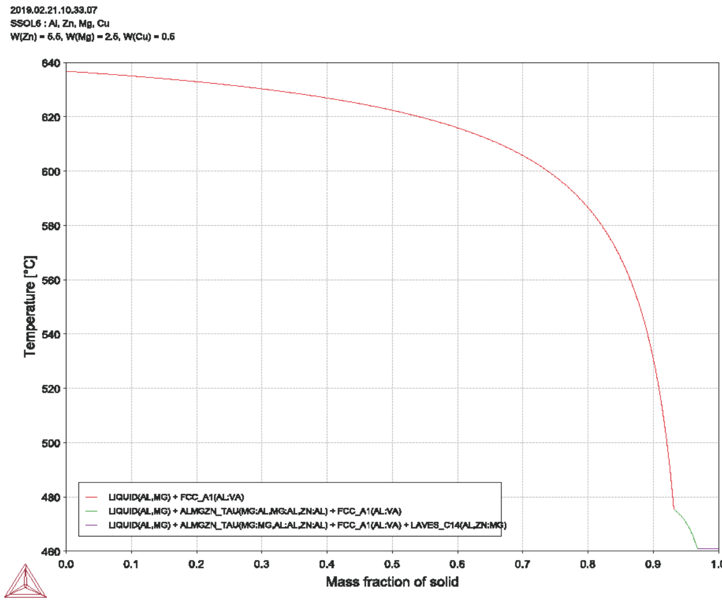
Creep tests were also conducted with a nano-indenter to analyze the time-dependent responses of the metal matrix composites prepared within the scope of the study. These tests with a Berkovich indenter used and 25 indents were created on a 5x5 grid in each test specimen. These formed indents were placed along the edges of the grid at intervals of 50 μm and 75 μm, and the load applied during the test was increased to a maximum load of 50 mN at a speed of 5 mN/s, keeping this load value for 500 s, and then unloaded. Immediately after this test process, the modulus and nano hardness values were calculated during the unloading phase of the creep test.

3. Results and Discussion

3.1. Comprehensive Microstructural Analysis

The result of Differential (Dynamic) Scanning Calorimetry (DSC) for Alumix-431, which is used as a component of the matrix to identify critical conversion points in the heating and cooling phase (in order to observe the relevant phases clearly, the heating and cooling rates were as low as possible,~ 2°C/min, under inert N_2 atmosphere) within the scope of the study, is given in Figure 1. Furthermore, a general microstructural view for the sintered specimen is presented, however.

A relatively homogeneous dispersion of reinforcement elements in the matrix was observed, it was determined that a very tough structural relationship was formed between these interfaces due to a good chemical bond diffusion at the interfaces of the matrix-reinforcement elements.



(a)

(b)

Fig.1 (a) DSC diagram measured for Alumix-431 and (b) SEM general microstructure for the sintered specimen

Figure 2 shows a microstructure taken from a sinter + forging specimen with "EDS" chemical analysis obtained on the "SEM" with BSE (Back Scattered) option. The reinforcement effect on the microstructure seems well and some of the areas show eutectic reaction due to chemical diffusion bonding. This is basically due to the easy diffusion of copper in the matrix. It seems that the reinforcements added to the composite were effectively improving its toughening mechanism. It is advantageous to add copper and γ - alumina for these types of composites, mainly because it provides a good combination of ductility and strength, which is useful properties for industrial applications during the manufacture of complex components. Therefore, the combined process (Sinter + Forging) always gives hybrid composites a very stable and robust microstructure.

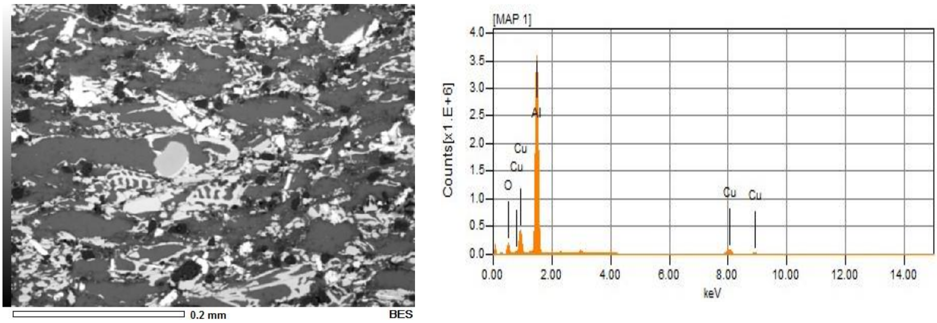


Fig. 2 Microstructure taken from a sinter + forging specimen with "EDS" chemical analysis obtained on the SEM with Back Scattered option

The distribution of the reinforcement elements in micro-sized very fine particles can be observed by "Mapping" analysis. As mentioned earlier on mapping analysis, it gives a safety observation of the homogeneity of the hybrid composite structure. The dispersion of reinforcement particles in the microstructure of the hybrid metal matrix composite for "MENG" produced with Sinter + Forging is given in Figure 3. Although the recycled chips used in the hybrid composite were atomized before the composite was prepared as a whole, the size of the chips varied between 10 and 200 µm. For this reason, the combined process called "sinter + forging" improves the dispersion of reinforcement particles within the final structure and increases the formation of a homogeneity and inner structure.

That is the reason we suggest this process especially for the recycled constituents as a low cost, alternative and more advantageous manufacturing process to design the composite [16-18].

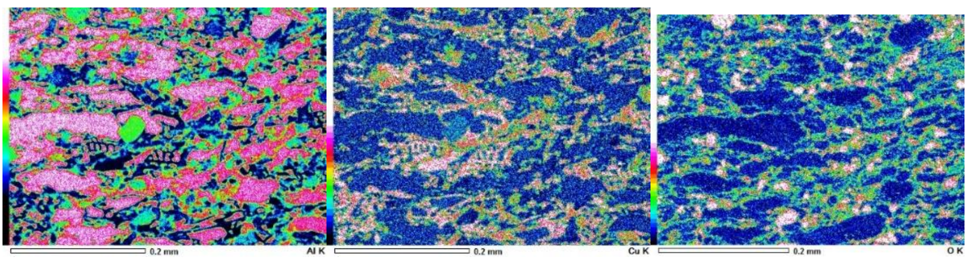


Fig. 3 Mapping elementary analyses of the hybrid metal matrix composite specimen (MENG, Sinter + Forging)

3.2. Quasi-Static Compression Test Results

In this research, quasi-static compression (DIN 50106) tests were performed under laboratory scales to evaluate the mechanical behaviour of these new hybrid metal matrix composites manufactured by sintering and sinter + forging manufacturing methods.

Quasi static compression test results of composite specimens obtained by sintering and sinter + forging manufacturing methods are shown in Figure 4. All quasi-static compression test results acquired for the combined method sinter + forging, were compared with the same type of test results obtained by the sintering process. These values obtained from the tests are demonstrated by taking the average values obtained from a minimum of 3 to 4 tests for each composite specimen. The standard deviation value in these experiments was determined to be at a variable value around ± 30-45 MPa. When the values given in Figure 4 are examined, it is observed that the sinter + forging process provides higher resistance compared to a simple sintering process.

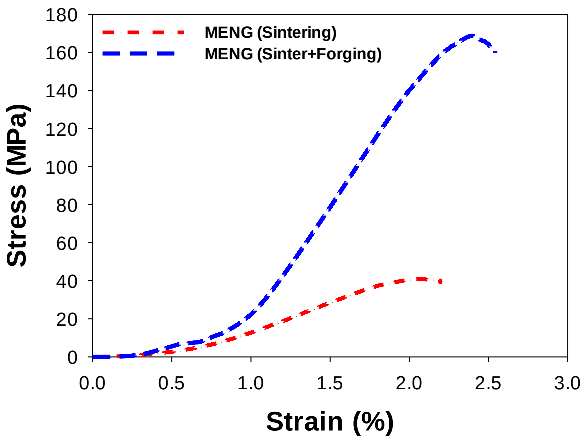


Fig. 4 Quasi-static compression test results for the sintered specimens and sinter + forging specimens

3.3. Three Point Bending (3PB) Test Results

As mentioned in the previous section of the study, these hybrid metal matrix composite parts were designed for connection / connector implementations in aerospace engineering, and for this reason, they were mainly studied in terms of bending forces under 3PB loading conditions. Figure 5 shows the 3PB (ASTM D 790) test results obtained for the sintered and sinter + forging specimens. All the other 3PB test results that were not presented here have indicated similar results with a variation of ±20 – 30 MPa. All the tests have been realized under laboratory conditions and they are mean values obtained from the repeat of 3-4 tests for each composition. According to the data in Figure 5, the yield strength values of the test specimens were determined as 55.8205 MPa for the MENG (Sintering) specimen and 89.9069 MPa for the MENG (Sinter + Forging) specimen. Again, the sinter + forging composite showed high toughness behaviour.

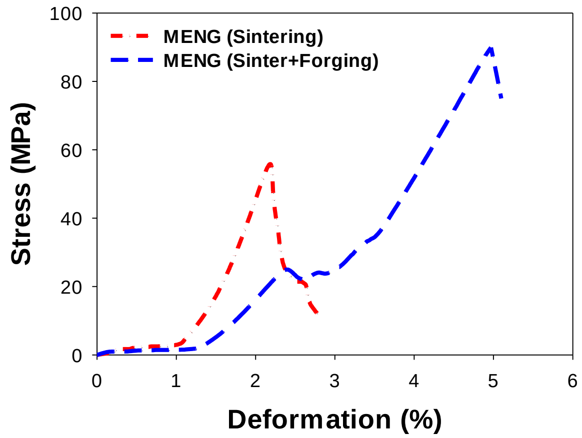


Fig. 5 "3PB" test results for the sintered specimens and sinter + forging specimens

3.4. Wear and Creep Test Results

For these tests, the creep compliance and the stress exponent were calculated by using data collection defined in Eq.1 [18]:

$$\varepsilon(t) = \sigma_0 J(t) \tag{1}$$

Where σ_0 is the constant stress applied and $J(t)$ *is* calculated using Eq.2

$$J(t) = A(t)/(1-\nu)P_0 \tan\theta \tag{2}$$

Where; A(t) contact area; P_0 constant applied load; is expressed in Equation 2 where the effective cone angle is 70.3 ° for a Berkovich indentation and the Poisson ratio (ν) is 0.3.

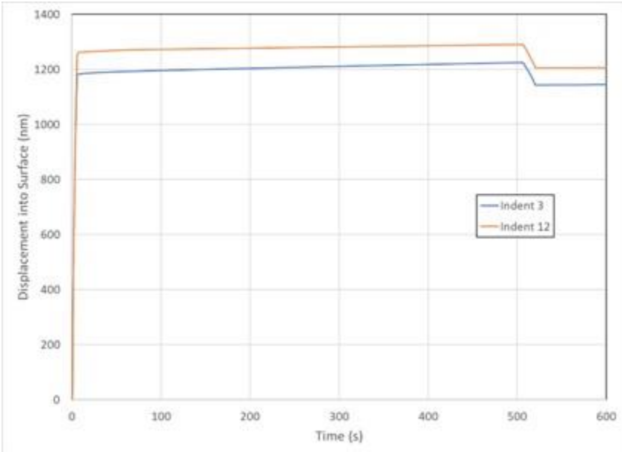
Thanks to this approach, it takes into account the change in the contact area under the Berkovich tip caused by displacement towards the surface.

The strain versus time behavior during creep is characterized by a high strain rate in the primary phase of creep. The secondary stage has a constant creep state given the strain rate given in Equation 3.

$$\dot{\varepsilon} = K\sigma^n \tag{3}$$

Where, K is constant and n is the stress exponent. The strain rate is calculated using software and n is obtained from a log-log plot of strain rate versus stress in the secondary phase of creep.

Although the materials investigated within the scope of the study were tried to be created in a homogeneous structure as much as possible, the microstructure was heterogeneous. It was observed that the nano-indentation test was performed on a small area / volume, causing a large scattering in the test result data. In order to come through this situation, the specimen number was taken as much as possible. Figure 6 shows the displacement time plots for indents 3 and 12 on the sinter + forging specimen. A picture of the indents was also presented in Fig 6. These curves are typical for the other indents on both sinter and sinter + forging specimens in which no significant creep was observed, and hence the creep exponents were not calculated.



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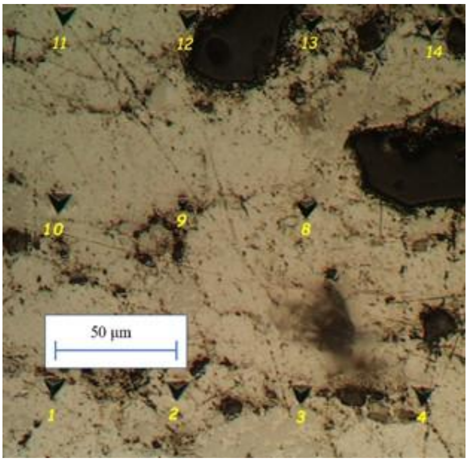


(b)

Fig. 6 (a) Typical displacement into surface vs time curves during creep tests and (b) Micrograph of the indents

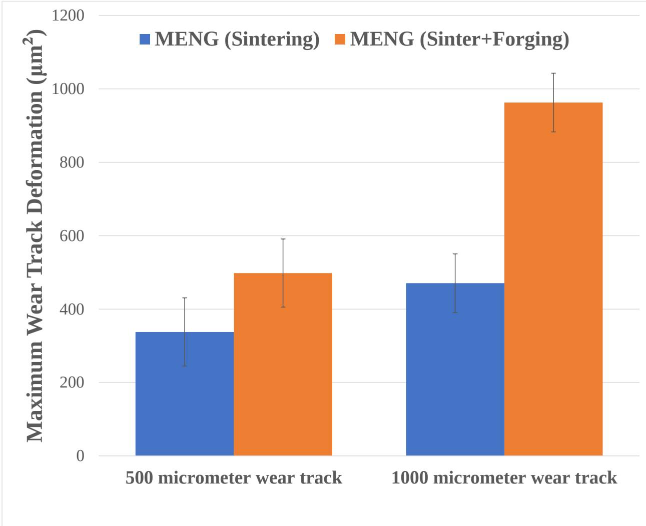
The modulus and hardness of the composites as measured during the unloading phase of the creep tests and their standard deviations are presented in Table 4.

Table 4. Modulus and hardness from unload data

Composition Name	Modulus (GPa)	Hardness (GPa)
MENG (Sintering)	77.15 ± 14.69	1.81±0.63
MENG (Sinter+Forging)	91.78 ± 11.79	1.81±0.86

Figure 7 depicts the maximum wear track deformation measured as the area between the initial profile and the final (residual) profile of the wear track (using Berkovich TB10157). These are the means of the 10 wear tests for each type of manufacturing. The sinter +

forging specimens resulted to be more resistant to wear. Also shown in this figure are 5 of the wear tracks for a 500 µm long wear path for the sinter + forging specimens.



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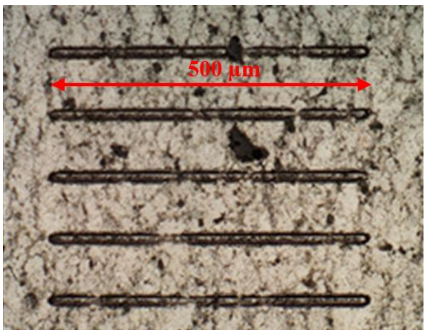

(b)

Fig. 7 (a) Maximum wear track deformation values obtained for the sintered and sinter + forging specimens and (b) Micrograph of 5 of the residual wear tracks for 500 µm wear tracks for the sinter + forging specimen

3.5. Low Velocity Impact Test Results

The results of the low velocity impact test for specimens manufactured by sintered and sinter + forging combined methods are shown in Figure 8. During the test, the reactive maximum forces on the hybrid metal matrix composite specimens were analyzed using the values obtained from both support data points. In this context, as circumstantiate in the section 2, a series of impact tests were performed by using a low velocity impact test (dynamic compression or instrumented drop weight test) device and applying instant force from the center (under room temperature) to the prepared cylindrical composite specimens through the impactor tip. The tests were repeated on three or four different specimens to determine the average values of the results obtained from the tests performed to determine the low velocity impact behavior of the hybrid metal matrix composite composition.

First of all, the results obtained from the tests, the integrated effect of the sinter + forging process on the impact resistance of hybrid metal matrix composite specimens were evaluated (Fig. 8). These graphs given in Figure 8 express the higher impact force damping and / or absorption capacity of sinter+forging specimens than specimens manufactured by simple sintering process. This indicates that the impact resistance of the composite specimen is directly related to how much of the energy produced during the test can be absorbed. All specimens tested with the low velocity impact test under laboratory conditions show that most of the impact force is used to balance with the inertial force. Also, only an undersize fraction of the impact force is used to damage through deformation and / or cause the specimen to break. The energy absorbed by the composite specimen must be correlated with the production process used during the manufacturing of the specimens. In this respect, when the specimens were examined, the energy absorbed increases significantly in the specimens manufactured by the sinter + forging method. Since these results obtained from the tests are reached at laboratory scales, they need to be developed and improved with detailed analyzes for industrial applications [8, 16, 18].

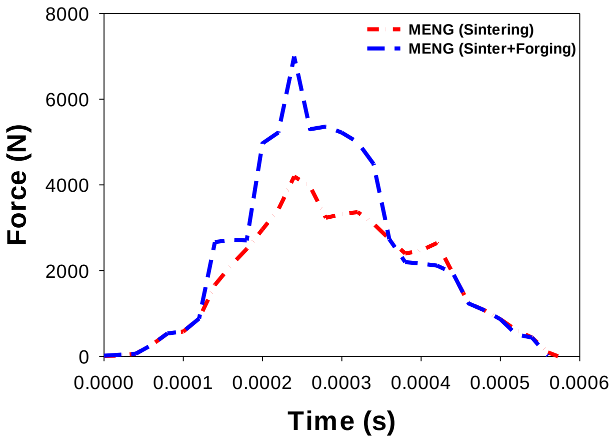

Fig. 8 Force (N)-Time (s) for the sintered specimens and for the sinter + forging specimens: Low velocity impact test results

4. Conclusions

A new hybrid metal matrix composite material (aluminum based) has been improved using fresh scrap aluminum with recycled Alumix-431 (50% wt) and AA1050 (50% wt) chips for aerospace / automotive connection components. These new hybrid composites have been successfully manufactured by both sintering and combined sinter + forging methods as low-cost manufacturing and necessary mechanical tests have been carried out. The addition of copper reinforcement together with γ-alumina can give a good combination of high ductility and high strength [5, 9, 13-18]. The sinter+forging process is a manufacturing process that is defined as a process of forming close to high accuracy for the manufacturing of parts processed from fresh scrap materials in the literature [6-14].

In the microstructural analysis, it was determined that the hybrid composite specimens manufactured by the sinter + forging combined process had a better chemical bonding diffusion at the matrix-reinforcement interface. In the microstructure of these composites, they showed a tough and complete microstructure feature that does not contain porosity.

The doping process and good powder mixture preparation conditions must definitely be improved in order to further improve the microstructure and further increase the wear resistance and ductility values. That is, a longer time of ball milling is required to contribute to a finer and more homogeneous dispersion of particles in the matrix.

In this current research, novel composite reinforced with γ-alumina and copper was designed for possible use in linkage/connector parts in such an aeronautical application [7, 11, 16]. This combined manufacturing process (Sinter + Forging) is considered to be a very safe approach for future studies of manufacturing alternative parts used in mechanical / electronic-like fastening components used in the aeronautical / automotive industry, as well as in other tribological applications. Optimization of operational parameters of production methods requires much more experimental research to make industrial scale real parts.

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