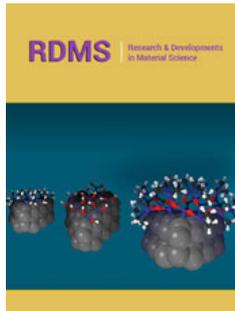


Mechanical Characterization of Graphene/ Graphite Added 7075 Composites after Sintering and Thermomechanical Treatments

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Abstract

The aim of this work is to develop 7075 based grafen/graphite added metal matrix composite for different applications. A series of aluminum based P/M MMC's alloys were prepared with grafen and graphite addition to 7075 based P/M alloys were mixed with %1 grafen powder and pressed and sintered at vacuum condition. After sintering hot swaging were carried out heating specimens at 350 °C in a resistance furnace. The tensile strength values of the consolidated bars after T6 heat treatment is in the range of 345-431MPa inspite of local agglomerations in the alloy matrix. Wear depth measured at 10N load for graphene added composites revealed 150µm wear which is less than graphite added composites have 250µm measured value.

The aim of this reseach is to develop graphene/graphite added AA7075 alloy matrix composite material for different applications such as piston ring bearing to improve solid lubrication. A series of 7075 composite powder alloys were prepared with 1% graphene were mixed, cold pressed and sintered under vacuum condition. After sintering hot swaging experiments were performed preheated at 350 °C in a rezistance furnace deforming flat cross sections into round bars. T6 heat treatment were applied to hot swaged specimens. Although the composite microstructures consist of local agglomerations in the matrix. Tensile strength values measured at room temperature are in the range of 345-431MPa.

Among the same group specimens tested at 10N load wear depth measured for graphene added composites is 150µm but graphite added composites' wear depth measured is 250µm. In other words graphene added ones revealed less wear compare to graphite added 7075 matrix composites.

Keywords: Powder aluminum alloy; Graphene; Graphite; Hot forging; Swaging; Vacuum sintering

Introduction

There is need for development of wear resistant pistons with modified ring and crown sections against high temperature fatigue and creep during service period of internal combustion engines.

By adding graphite/graphene solid lubricant materials into 7075 aluminum powder composite placed near to ring section and local reinforcement of crown front by SiC particles as a strategy of insertion functionally graded materials to improve high temperature fatigue resistance and SiC particles in the matrix to prevent crack propogation in crown sections expose to combustion chamber of the pistons.The functionally graded insertions can be created and produced by sintering.

Sintered aluminum matrix composites are planned to be placed in two diffent sections of the piston cavity then pressure infiltration casting is to be applied to the permanent mould to integrade and finish processing. Racing car and manless aircraft engines requires ceramic particle reinforced sections and pistons having prolonged service life due to higher fatigue

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and wear resistance with higher combustion temperatures and better fuel efficiency.

The need for higher efficiency and high compression pressure in the internal combustion engine still required in hybrid solutions for electric powertrains. Better combustion efficiency and performance is starting point of research on the development of pistons with functionally graded composite inserts. Aluminum matrix composite rings are under development stage to replace iron based rings with aluminum based ones which are more expensive, limited and lighter than traditional solutions.

One of the main goal of this work is develop an economical technique for 7075 graphene/graphite added composite material for insert of ring bearing sections by sintering processing and characterization. Graphite and graphene based materials used in pistons are planned for weight saving and solid lubrication to decrease friction coefficient. Powder handling stage of the work start with mixing of pure Al, Zn, Mg and Cu powders then sintering under vacuum condition. Alloy was prepared by melting and alloying in induction furnace.

When liquid phase and alloying exist two major phases α -Al and $MgZn_2$ are equilibrium phases yields a eutectic at room temperature. The other possible phases forming during solidification are

$S-Al_2CuMg$, $T-Al_2Mg_3Zn_3$, and Al_2Cu phases($Al-Zn-Mg-Cu$) [1,2] The ratio of Zn to Mg ratio is effective in $MgZn_2$ intermetallic formation. High zinc to magnesium ratio result in the formation of other phases than $MgZn_2$. These phases in nano and micro size form in the alloy matrix after T6 heat treatment. They reinforces the alloy matrix.

Materials and Methods

In this work to make 7075 composites Al, Zn, Mg and Cu element powders were mixed and sintered. Graphene powders were produced by Sintermetal company using cryomilling technique for more than 48 hours. The carbon base samples were tested by a Raman Spectrometer to identify cahacteristic peaks at METU Restoration laboratory in Faculty of Arcitecture. After identification of the graphene powders they were mixed with Al, Zn, Mg, Cu and powders. Sintered preforms were prepared using Al, Mg, Zn and Cu element powders mixing, compacting and sintering under controlled atmosphere furnaces in Sintermetal company's industrial process facilities. The four different group of powder preforms are given as:

1. Sintered 7075 plain mixture
2. Sintered 7075+%1 graphite added
3. Sintered 7075+%1graphene added 48hrs milled
4. Sintered 7075+%1 graphene added (China)

After cutting the sintered rectangular shapes into square cross section bars(50x10x100mm) they were hot forged under 100 Tons hydraulic press at METU Metal Processing Laboratory.

Second group specimens were hot swaged using FENN swaging machine at METU workshop (Figure 1).



Figure 1: Automated 100 Ton hydraulic press used for hot forging experiments at METU.

Hot forging experiments of 7075 sintered specimens

1. First group specimens initially solutionized at 485 °C for 3 hrs, water quenched and aged at 120 °C for 12 hrs then preheated to 250 °C for hot forging.
2. Second group specimens initially solutionized at 485 °C for 3 hrs, cooling to 350 °C then hot forged. Finally aged for 12hrs.

The tensile test specimens of the first two group specimens in were machined from flat rectangular shapes of hot forged slab blanks. Four groups of sintered specimens in thin slab shape were solutionized and water quenched, machined into standard tensile test geometry at T0 condition then heat treated for T6 condition by ageing at 120 °C for 12hrs. Tensile tests were performed at room temperature..Thermomechanical process were applied to all 7075 sintered specimens. First two group series processed by hot forging and second two groups sintered specimens were processed by hot swaging. Hot forged specimens are machined in flat form but hot swaged specimens were machined into round cross section.

First group T6 heat treated and hot forged specimes were machined into flat shape and tested by Instron tensile test unit at METU mechanical testing laboratory (Figure 2).

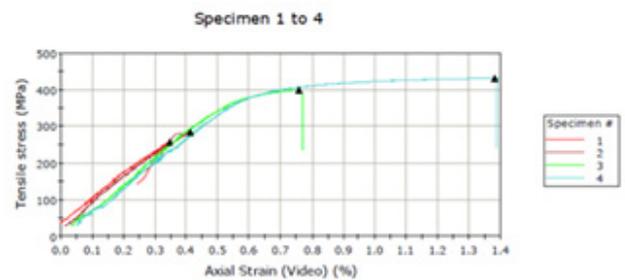


Figure 2: First group AA7075 consist of hot forged slabs machined into flat tensile test specimens.

3. Hot swaging experiments were carried out at METU Metallurgical and Materials Engineering Department's work shop by preheating at 350 °C in a resistance furnace then swaging in FENN machine reducing the cross section from 12mm into 8mm diameter round bar shape. T6 heat treatment was applied to the rods obtained (Figure 3).



Figure 3: Machined round tensile test bar specimens form hot swaging operation and heat treated for T6 condition. The tensile test results are given in Table 1.



Graph 2

Figure 4: Highest tensile strength values of hot swaged samples after T6 heat treatment.

The specimens labeled as 2-4, 3-3 and 4-2 tensile strength values are in range of 350-431MPa but percent elongation is not more than 1.4. This is due to an even distribution of graphene/graphite in the matrix, microporosity and unreduced oxides along grain boundaries (Figures 5-7).

Results and Discussion

This table shows thermomechanically processed sintered and hot forced 7075 alloys having highest strength and hardness values (Table 1).

Table 1: Tensile strength and microhardness after sintering and hot forging of two highest values form each group.

7075 HF GR1-1	Tensile strength 350 MPa	HV 144
7075 HF GR1-4	Tensile strength 257 MPa	HV 149
7075 HF GR2-1	Tensile strength 313 MPa	HV 135
7075 HF GR2-2	Tensile strength 345 MPa	HV 137

After sintering and hot swaging experiments highest values selected from four groups are given in Table 2.

Table 2: Tensile strength and microhardness of sintered and hot swaged samples.

7075 HSW 1-4	Tensile strength 431 MPa	HV 149
7075 HSW 2-4	Tensile strength 354 MPa	HV 135
7075 HSW 3-3	Tensile strength 313 MPa	HV 138
7075 HSW 4-2	Tensile strength 345 MPa	HV 146

The tensile strength values of hot swaged specimens are higher than hot forged specimens. Tensile strength value of the specimen 1-4 is 431MPa and there is no flake like agglomerations in the matrix and graphene distribution is more homogeneous. As a common observation when microhardness measured to be high corresponding tensile strength values are also high (Figure 4).

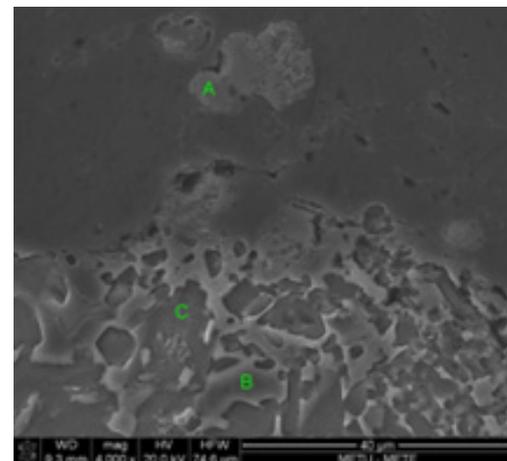


Figure 5: Hot swaged 7075 HSW 1-4 SEM image and point analysis A(Al,Fe,Cu,Si,Zn), B(Al,Zn,Cu,Mg) C(Al,Zn,Cu,Cr,Mg). Tensile strength: 431 MPa.



Figure 6: The optical micrograph of hot swaged specimen after T6 heat treatment. HSW 1-4 Huvitz optical 3D microscope image. Tensile strength: 431MPa. Black regions are graphite/graphene rich zones.

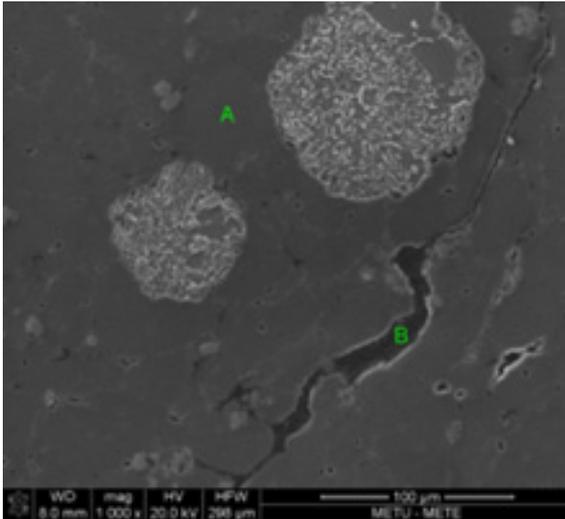


Figure 7: Hot swaged 7075 HSW 3-3 SEM image A(Al,Zn,Cu,Mg), B(C, Al, Si) Tensile strength: 313MPa.

The appearance of carbon rich region marked as B which is flake shape. This result reveal the mixing difficulty of graphene powders into 7075 aluminum powders due to surface tension and surface activity of ultrafine powders. There is tendency of segregation of graphene powders during mixing stage. To overcome this difficulty mixing period was extended to 45 mins. The point A stands for matrix and Zn, Mg and Cu peaks reveal the effective homogenization of premixed powders at sintering stage.

The precipitate phase reflections of $AlMgZn$, $AlMg_2Zn$ and $MgZn_2$ were reported beside Al peaks in sintered samples given in the literature. Mg_2Zn_{11} phase was also reported in the thermomechanically processed 7075 alloys [3-5].

Wear testing of the samples

After sintering and four group 7075 alloy samples in rectangular shape were tested applying 10 and 20N loads by UTS Tribometer T10/20 in Iron and Steel Institute of Karabük Universtiy (Figure 8). The wear depths of sintered 7075 specimens prepared at Sintermetal facilities and tested at Iron and Steel Institute Karabük University are given below (Figures 9-12); Table 3.

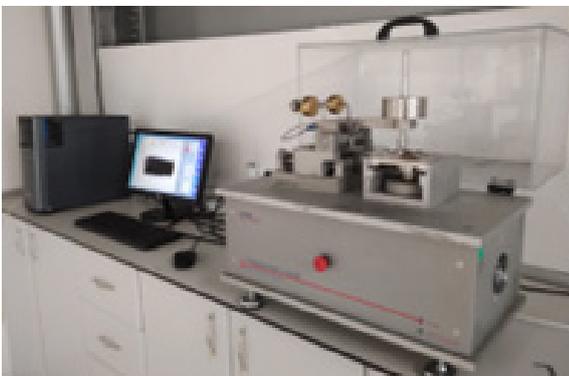


Figure 8: Wear test device used for testing of specimens in Iron and Steel Institute Karabük University.

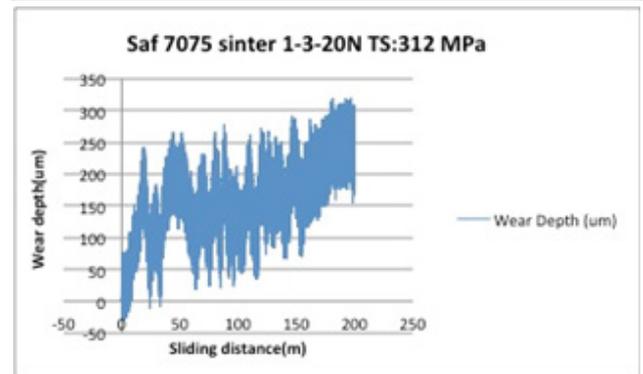
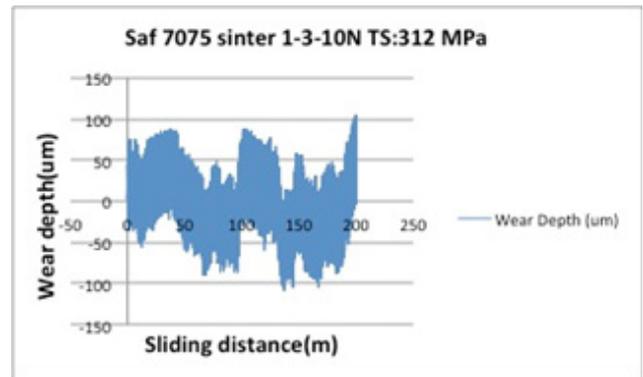


Figure 9: Plain 7075 sintered specimens tested under 10N and 20N with steel ball movement on the surface of aluminum samples with back and forward action.

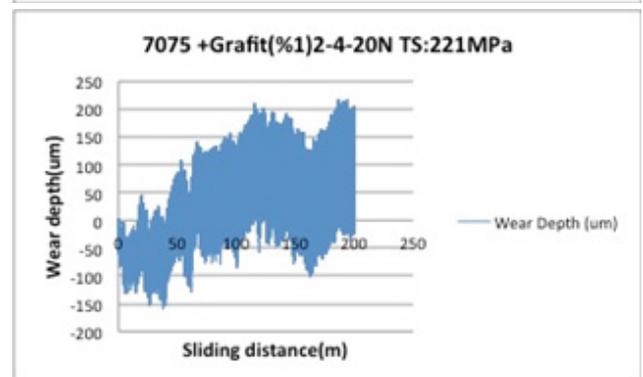
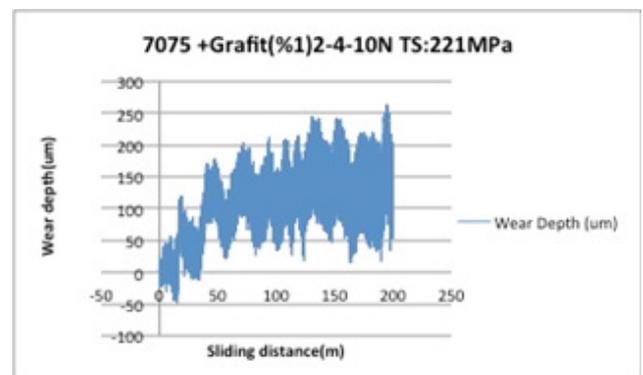


Figure 10: 7075+Grafıt(%1) sintered composites tested under 10N and 20N load wear testing with back and forward moving action.

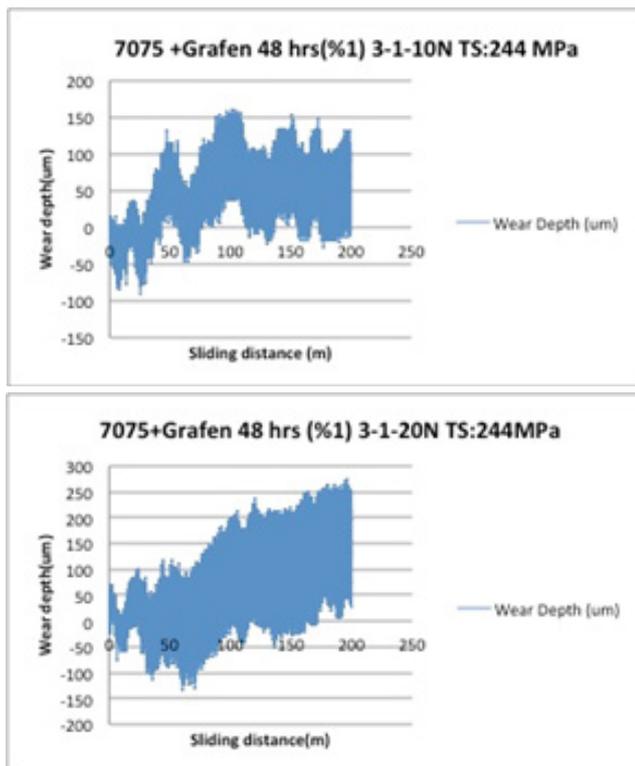


Figure 11: Plain 7075+Graphene 48hrs(1%) sintered composites tested under 10N and 20N load wear testing with back and forward moving action.

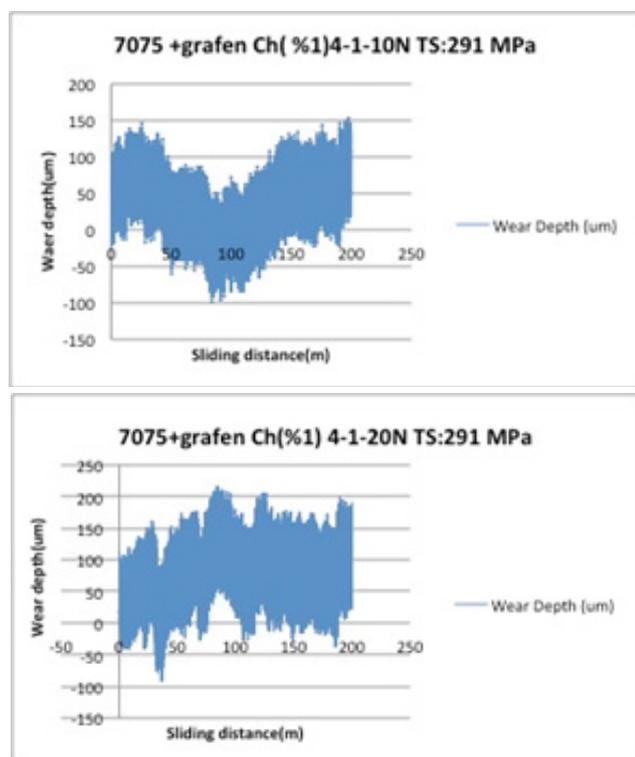


Figure 12: 7075+Grafen China(1%) sintered composites tested under 10N and 20N load wear testing with back and forward moving action.

Table 3: The results of wear testing of four 7075 group sintered and thermomechanically processed powder alloys.

Alloy Load(10 N)	Wear Depth (µm)	Friction Coefficient	Friction Force
Plain sinter 7075	75-10	1-0.8	20-18
7075-graphite(%1)	50-250	1.5-1.7	28-30
7075-graphene 48 hrs (%1)	0-150	1-1.5	20-28
7075- graphene China (%1)	100-150	1.0-0.8	20-15

Alloy Load (20N)	Wear Depth (µm)	Friction Coefficient	Friction Force
Plain sinter 7075	250-300	2.2-1.8	40-39
7075 graphite (%1)	0-200	2-2.5	40-55
7075 graphene 48 hrs (%1)	50-250	2.2-1.8	40-38
7075 graphene China (%1)	100-200	2-1.5	40-30

The tensile strength and microhardness values increase with a positive trend after hot forging and swaging operations. The reason for this trend is the formation of nano-size $MgZn_2$ precipitates after T6 heat treatment, which cannot be identified by SEM analysis. After sintering, thermomechanical processing, and T6 heat treatment, hot swaged samples possess higher tensile strength values than hot forged ones.

Tensile strength values of hot forging after sintering and T6 heat-treated samples are 350 MPa, whereas the tensile strength of hot sawing and T6 heat-treated samples is measured as 431 MPa. The possible intermetallic phases reported in the literature are Al_2CuMg , $T-Al_2Mg_3Zn_3$, and Al_2Cu phases (Al-Zn-Mg-Cu) [1,2]. According to the SEM examinations performed on the specimens produced in this work, revealed $MgZn_2$ besides copper-containing Al_2CuMg phase. Since these phases exist together, point element analysis performed by EDX analysis shows Al, Zn, Mg, and Cu peaks in the same spectrum.

Wear depth measured for the first group of plain 7075 sintered samples tested under 10N load is increasing from 75-10 µm to 250-300 µm range. Wear depth measured under 10N load for graphite-added group is 250 µm, and graphene-added group is 150 µm. As a summary, graphene-added composites show less wear than graphite-added composites. The same trend was observed in friction coefficient measurements given in Table 3. Friction coefficient determined under 10N load for graphene-added group is in the range of 0.8-1.5, and graphite-added group is in the range of 1.5-1.7, which is higher than graphene-added composites.

In the second series of friction tests measured for plain sintered 7075 under 20N, the highest wear depth is increasing from 75-10 µm to 250-300 µm range. Graphite-added group shows an increasing trend from 50-250 µm range to 0-200 µm range. Graphene (48hrs) group showed an increase from 150 to 250 µm, and graphene (China) revealed an increase from 150 to 200 µm under the same loading condition. Friction coefficient of plain 7075 alloy under 20N load drastically increases in range from 1-0.8 to 2.2-1.8, but

graphite added group increases from 1.7 to 2.5, graphene 48hrs group increases from 1.5 to 2.2 and graphene(china) added group increases from 1.0 to 1.5-2.

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