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Effects of disc milling and indirect extrusion processing on mechanical properties of aluminum-graphene-composites with commercial GNPs

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Abstract. In this study aluminum-graphene-composites were manufactured applying a powder metallurgical approach. After graphene exfoliation and mechanical mixing, powders were disc milled, compacted and extruded into round rods. Two different sources of commercial graphene nano platelets (GNP) (by 1. Sigma Aldrich (SA) and 2. Alfa Aesar (AA)) were investigated with the main difference being their specific surface area. One subject of the study was to investigate the effect of disc milling processing on the mechanical properties of Al/GNP composites. Results indicated that micro hardness as well tensile yield strength (TYS) and ultimate tensile strength (UTS) were not improved significantly or only slightly when SA-GNPs and a disc milling duration of 1min were applied. Varying the extrusion ratio from R=9:1 to R=14:1 and R=31:1 as well as applying conic or flat face dies for extrusion did not have a significant effect on the mechanical properties. But when disc milling duration is extended to 5min, significantly increased micro hardness (up to 30%), TYS (up to 17%) and UTS (up to 26%) were observed. Application of AA-GNPs as a second source of commercial GNPs also resulted in significantly increased micro hardness (up to 49%), increased TYS (up to 16%) and UTS (up to 27%) when disc milling of 5min is conducted in the powder processing stage. SEM analysis indicated that after 5min disc milling aluminum particles were deformed from spherical into a plate-like shape. Additionally, GNP exfoliation and its dispersion in the Al matrix seemed to be improved by the longer milling duration and could be a reason for the enhanced strengthening effect of GNPs.

Introduction

Graphene is a 2-dimensional carbon-based material ideally consisting of planar carbon monolayers, where carbon is sp^2-hybridized. It was first synthesized successfully in 2004 [1]. Due to exceptional high intrinsic strength and elastic modulus [2] monolayer graphene is highly attractive to be applied as strengthening additive for composite materials such as aluminum-graphene-composites. But due to high van der Vaals forces, graphene layers tend to agglomerate which is detrimental to the mechanical composite properties [3]. Thus, graphene exfoliation and its homogeneous dispersion in the matrix material are key for achieving a high strengthening effect. Furthermore, the formation of carbides can be a reason for decreasing strength and ductility [4]. The current study investigates a processing approach of graphene sonication (for exfoliation), disc milling, cold compaction and indirect extrusion to manufacture aluminum-graphene-nanoplatelet(GNP)-composites. In a prior study, aluminum and graphene powders were mixed mechanically only, then compacted and extruded. This approach did not lead to significant strengthening. The main reason for that was assumed to be GNP agglomeration, as GNPs were found to be aligned in relatively thick layers around aluminum particles, especially for high GNP contents [5]. Thus, for the current study disc milling was added to the process chain. Disc milling is a method usually applied to reduce the size of particles through compression, shearing and friction forces. Thus, with disc milling a high amount of energy can be introduced into the powder mixture, aiming to decrease the GNP agglomeration around aluminum particles and improve GNP dispersion. Furthermore, in this study two different commercial graphene sources were applied and the resulting mechanical properties of the Al/GNP composites are compared.

Materials and Methods

In this study Al/GNP composites were prepared via a powder metallurgical route. Same pure (99.7%) aluminum powder as in a previous publication [5] was applied as matrix material. Aluminum with mean particle size of 49µm was provided by TLS Technik GmbH & Co. Spezialpulver KG, Germany. In the first part of this research the effects of GNP-content, extrusion ratio, die angle and milling duration on micro hardness and tensile properties were investigated exemplarily. In order to study the general relationships between processing parameters and the material properties for each condition one rod was extruded. Micro hardness was tested on one rod cross section extracted from the middle length of each rod, three samples were cut from the middle to end region of the rod and tested in tensile tests. For the first part of the study GNPs provided by Sigma Aldrich were applied. All extrusion experiments were carried out on a small scale 0.5MN extrusion press with a container of 30mm diameter at Extrusion R&D Center TU Berlin. Billets were prepared by first GNP exfoliation through sonication (Branson Sonifier 450) in ethanol for 10min. The GNP/ethanol solution was then added to aluminum powder in an Eirich intensive mixer and batches with GNP-contents of (0.25%), 0.5% and 1.0% were prepared. Mechanical mixing was performed for 360s at 1800rpm. Subsequently powders were dried in a vacuum furnace for 16h at T=50°C. Powder mixtures were then disc milled applying the milling tools given in Fig. 1 rotating at 1000rpm for 1min and exemplarily for 5min. Milling batch size was 100g, respectively. Due to significant heat generation overall milling duration of 5min was split into 5 steps of 1min each with cooling breaks of a couple minutes in between. The weight of the tools (2 rings and 1 disc) rotating within the milling die was 9.2kg (Fig. 1). Milled powders were then sieved with mesh size of 172µm and subsequently cold compacted with 200MPa into solid discs for better specimen handling. Compacts were then extruded at 300°C in indirect extrusion mode applying different extrusion ratios of R=9:1, R=14:1 and R=31:1. Additionally, two different die angles (2〈=180° flat face die and a conic die with 2〈=130°) were applied for selected samples to investigate its effect on the composites' properties, exemplarily.

In the second part of the investigation an alternative graphene source, provided by Alfa Aesar, was applied. The Alfa Aesar (AA)-GNPs especially featured a significantly higher specific surface area of 529m^2/g than the Sigma Aldrich GNPs (108m^2/g). In order to characterize their qualities both graphene sources were investigated by Raman spectroscopy. The effect of AA-GNP content as well as the effect of powder mixing or mixing with additional disc milling for 5min on the mechanical properties was studied. Extrusion of the 5min disc milled material with AA-GNPs was carried out applying the conic die (2〈=130°) with diameter 8.0mm (R=14:1). Effects of extrusion ratio, milling duration and die angle variation were not investigated for AA-GNPs.

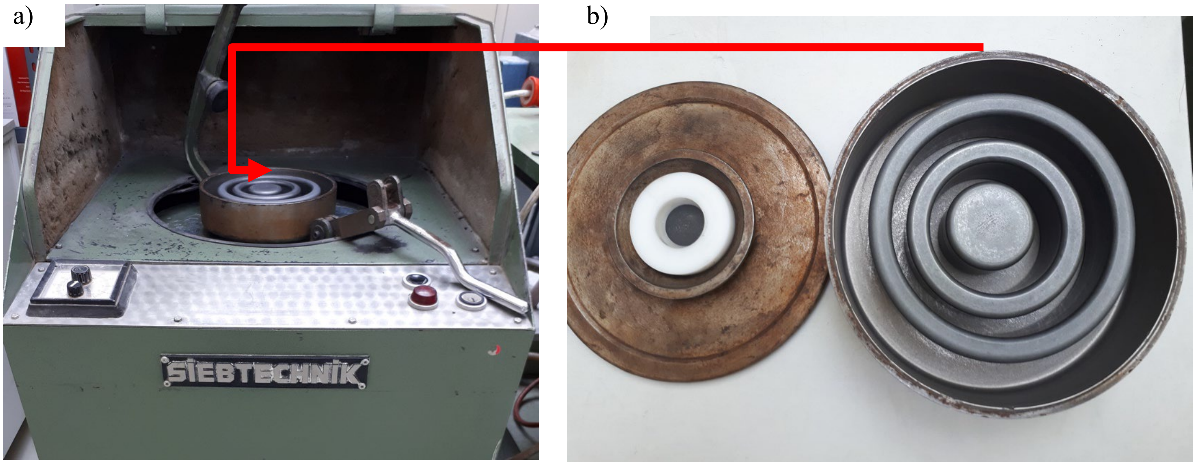

Fig. 1. a) Disc mill b) milling die with two milling rings and one disc.

Results and Discussion

Powder characterization.

In a first step the initial powders were investigated via SEM in as received condition. Results are displayed in Fig. 2. The pure aluminum powder is given in Fig. 2a. The powder predominantly is of spherical shape with varying size between 5µm and 70µm. Mean particle size was 47µm. Fig. 2b presents the GNPs provided by Sigma Aldrich. These GNPs were mostly found attached to each other and had a very flat shape with sharp edges. In contrast, the GNPs provided by Alfa Aesar consisted more of spheric particles with very fine platelets on their surface (Fig. 2c).

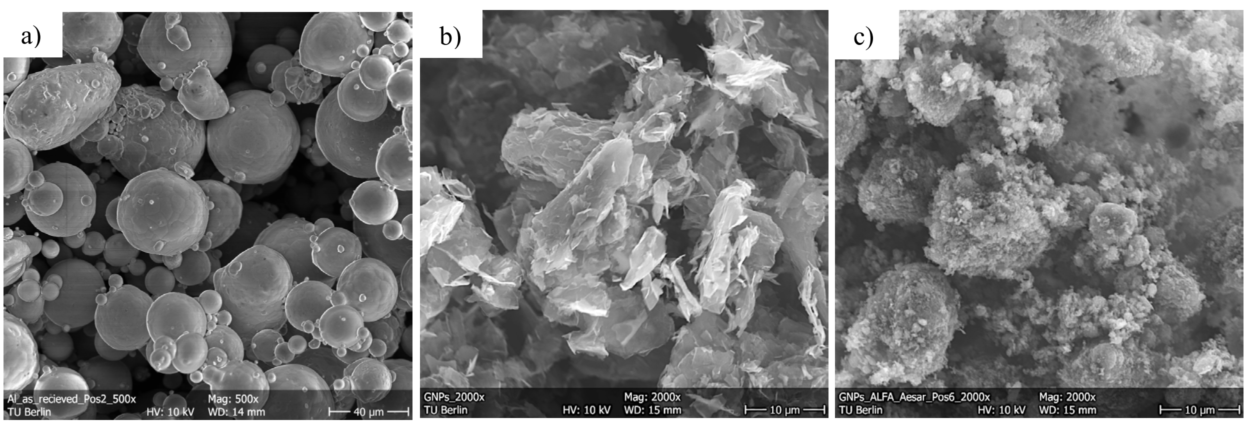

Fig. 2. SEM images of as received a) pure Al powder (99.7%), b) Sigma Aldrich (SA)-GNPs c) Alfa Aesar (AA)-GNPs.

In order to investigate the effect of each powder processing step on morphology and GNP dispersion, the composite powders with GNP content of 1% were selected exemplarily. SEM images are given in Fig. 3. Al-particles remained their initial spherical shape after mechanical mixing for 6min at 1800rpm as shown in Fig. 3a. SA-GNPs seemed to be relatively well distributed in the powder as no larger agglomerates were found. Although, often a few GNPs were stacked on top of each other. After 1min of disc milling first effects of the high energy input due to the milling process can be noticed (Fig. 3b). The surface of aluminum particles became rougher and their shape mostly was not as ideally spheric as in the initial state. After 5min of disc milling, due to the high impact energy through friction and shearing in the mill, aluminum particles were found to be deformed into plates with a rough surface (Fig. 3c). The results after 5min of disc milling seemed to be comparable to the deformation of aluminum powder after 1h of ball milling presented in [6]. Thus, applying disc milling instead of ball milling could be an option to produce composites since milling time and thus cost could be reduced significantly.

After mixing, the AA-GNPs were found to be attached to Al-particles in form of small chunks as indicated in Fig. 3d. But after disc milling for 5min they were found in a very thin, flat shape and reduced in size (Fig. 3e). Hence, it can be concluded that the high energy input throughout the disc milling process had a positive effect on the dispersion of AA-GNPs in the composite powder.

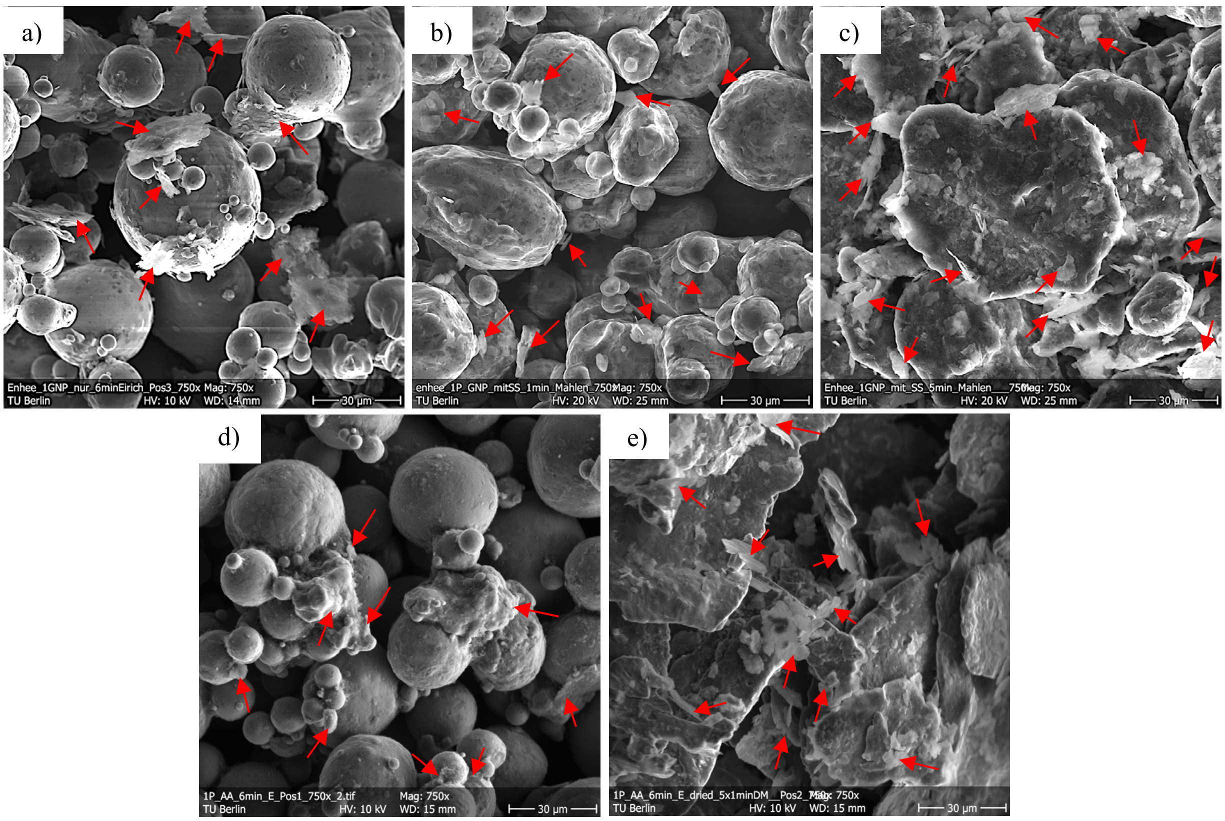

Fig. 3. SEM images of composite powders a) 1% SA-GNP after mechanical mixing, GNPs exemplarily indicated with arrows b) 1% SA-GNP after 1min disc milling c) 1% SA-GNP after 5x1min disc milling d) 1% AA-GNP after 6min of mechanical mixing e) 1% AA-GNP after 5x1min disc milling.

Raman spectroscopy is a fast, nondestructive method with high-resolution that can be applied for the characterization of e.g. lattice structure of carbon-based materials and thus provides information on the morphology and quality of GNPs [7]. The Raman spectra of the applied two different commercial GNPs are given in Fig. 4a and Fig. 4b. In both spectra the presence of the three prominent peaks namely D-band at ~1325cm^{-1}, G-band at ~1565cm^{-1} as well as 2D band at ~1650cm^{-1} were observed. The G-band corresponds to intrinsic in-plane C-C vibration modes of graphene and is characteristic for carbon-based compounds [8]. The D-band relates to the magnitude of defects or disorder in the crystal structure of graphene [9]. The 2D-band is related to number of graphene layers in the GNPs [10]. The defect density of GNPs can be predicted with the intensity ratios of D-band and G-band (I_D/I_G) and the number of graphene layers in the GNPs with the ratio of 2D-band to G-band (I_{2D}/I_G) [11].

Hence, the presence of both, G band as well as 2D band on the specific wavelength positions and the fact that the 2D intensity is much lower than the G-band (I_{2D}/I_G) leads to the conclusion that both GNP sources can be characterized as multilayer graphene. Furthermore, the ratio of I_D/I_G

leads to the conclusion that both GNPs contain defects and are disordered at least to some extent. By comparing the two GNP sources it can be stated that the AA-GNPs seem to be of better quality since the I_D/I_G ratio as a measure for defects and disorder is lower (Table 1). Additionally, the I_{2D}/I_G-ratio is higher for AA-GNP, which indicates that it consists of a fewer number of layers [10, 11]. Thus, both quality criteria were found to be in favor of the AA-GNPs.

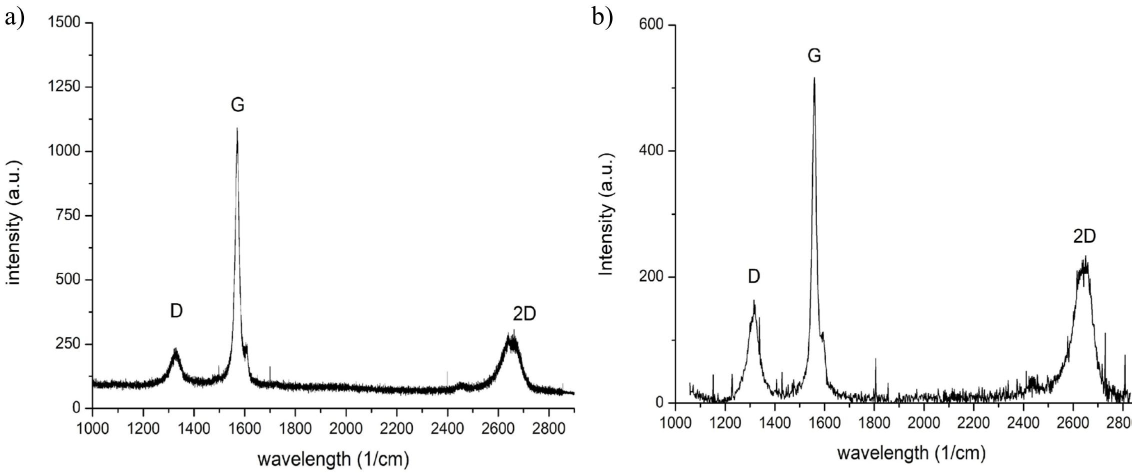

Fig. 4. Raman spectra for a) Sigma Aldrich GNPs GNPs b) Alfa Aesar GNPs.

Table 1. Results of Raman spectroscopy on both graphene sources.

	Sigma Aldrich (SA) GNPs		**Alfa Aesar (AA) GNPs**	
Peak	wavelength [1/cm]	Intensity [a.u.]	wavelength [1/cm]	Intensity [a.u.]
D band	1329	223	1317	159
G band	1571	1090	1560	515
2D band	2652	279	2649	258
I_D/I_G		0.80		0.62
I_{2D}/I_G		0.26		0.50

Characterization of mechanical properties.

Mechanical properties of the Al/GNP composites were characterized in terms of micro hardness and tensile testing properties in dependence of graphene content and relevant process parameters. The basic assumption for the effect of GNP-addition to the aluminum matrix material would be that if no agglomerates are formed, the mechanical properties of the composite should increase because of graphene's excellent mechanical properties.

Table 2a and Fig. 5a summarize the results of micro hardness testing on extruded composites with respect to the three SA-GNP contents (0%, 0.5% and 1.0%) as well as for the three different extrusion ratios (R=9:1, R=14:1 and R=31:1) when disc milling was conducted for 1min.

It can be noticed that the addition of SA-GNPs to pure aluminum did not lead to significantly increased hardness values. The only slight increase was observed for the 0.5%-SA-GNP-composite extruded at R=14:1. In that case hardness improved from 37.7 HV to 39.7HV (+5%). But a further increase of GNP content to 1.0% SA-GNPs resulted in a hardness of 37.3HV and thus, basically was the same as for pure aluminum. The effect of the extrusion ratio on micro hardness was limited as well but slightly more significant compared to the effect of SA-GNP content. For pure aluminum the effect was insignificant. But for SA-GNP contents of 0.5% and 1.0% an increasing tendency of micro hardness with increasing extrusion ratio could be observed (Fig. 5a). The highest effect was found for the 0.5% SA-GNP composites by extruding with R=14:1 instead of R=9:1. By that hardness was increased from 35.7HV to 39.7HV (+11%). In case of the 1.0% SA-GNP composites hardness increased by 7% when extruding with R=31:1 instead of R=9:1.

Table 2b presents the effect of extrusion die angle (2〈) on micro hardness. For pure aluminum hardness increased by 5% from 37.7HV to 39.5HV when a flat face die (2〈=180°) is applied instead of a conic one with 2〈=130°. For the composite material with 0.5% SA-GNPs the opposite effect was found as hardness decreased by 8% when the flat face die was applied instead of the conic one. For the 1% composite there was no effect at all. Hence, a clear systematic effect of the die angle on micro hardness was not observed.

Table 2c and Fig. 5b represent the hardness values of SA-GNP-composites after t_{DM}=1min of disc milling and after 5min (5x1min). Hardness of pure aluminum increased by 5% from 37.7HV to 39.5HV after 5min. As the Al-particles are deformed into plates at the longer milling duration (Fig. 3c & Fig. 3e), strain hardening is assumed to be the main reason for the increased hardness. In case of the 1% SA-composite the increase in micro hardness was 30% as it increased from 35.3HV after 1min to 45.7HV after 5min. This was the first and only real significant effect in all the micro hardness testing of SA-GNP containing composites. Hence, as presented in Fig. 3c the longer milling duration seemed to have improved exfoliation of SA-GNPs and maybe even decreased their size leading to a better SA-GNPs dispersion in the composite. Through its improved dispersion the graphene was more effective in increasing the micro hardness of the composite.

Table 2. Micro hardness of extruded composites containing SA-GNPs a) effect of extrusion ratio R and GNP content b) effect of die angle for R=14:1 after 1min disc milling c) effect of disc milling duration at R=14:1.

a) GNP content	R	HV0.5
0%	9:1	36.8±0.4
0.5%	9:1	35.7±0.4
1.0%	9:1	35.8±0.6
0%	14:1	37.7±0.6
0.5%	14:1	39.7±0.2
1.0%	14:1	37.3±0.4
0%	31:1	37.7±1.3
0.5%	31:1	38.1±0.2
1.0%	31:1	38.3±0.3

b) GNP content	die angle 2α [°]	HV0.5
0%	130	37.7±0.6
0%	180	39.5±0.6
0.5%	130	39.7±0.2
0.5%	180	36.4±0.2
1.0%	130	37.3±0.4
1.0%	180	37.3±0.6

c) GNP content	t_{DM}	HV0.5
0%	1min	37.7±0.6
0%	5min	39.5±0.6
1.0%	1min	35.3±0.5
1.0%	5min	45.7±1.6

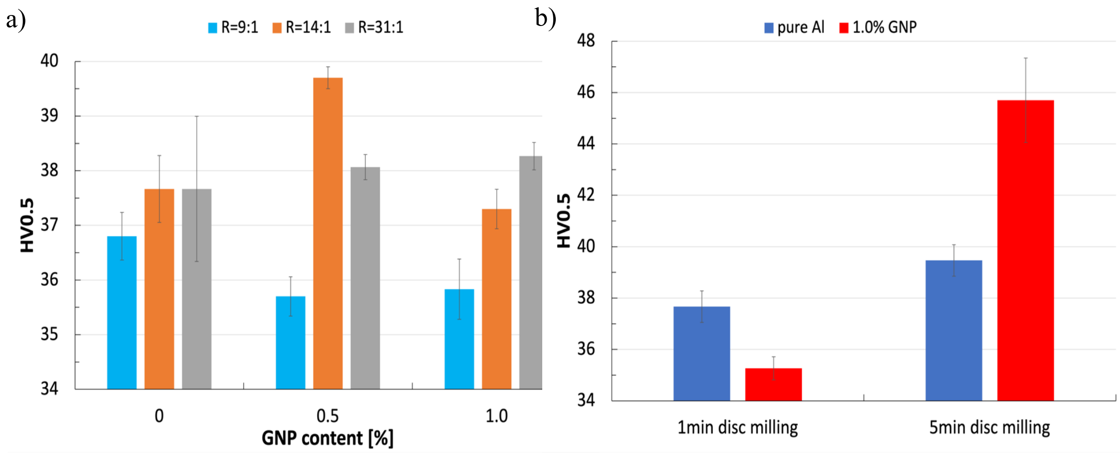

Fig. 5. Micro hardness of extruded composites containing SA-GNPs, a) processed via 6min mechanical mixing and 1min disc milling, extrusion at R=14:1 with conic die (2α=130°) b) effect of disc milling duration, applying R=14:1 and 2α=130° for extrusion.

Similar to the data representation of micro hardness, the results of tensile testing on the extruded SA-GNP composites are given in Table 3 and visualized in Fig. 6.

As observed before for the micro hardness, the tensile yield strength (TYS) did not change drastically with SA-GNP content nor with extrusion ratio. Values between 85MPa and 92MPa were measured for all composites that included disc milling for only 1min. The same can be stated for the ultimate tensile strength (UTS) since values varied between 110MPa and 116MPa. Even for the fracture strain no significant effect of the GNP content was measured. Values were between 25% and 29%. As can be noticed in Table 3b the effect of extrusion die angle (flat vs. conic) also did not have a relevant effect on the tensile properties of 1min disc milled SA-GNP composites.

Lastly, the effect of disc milling duration was investigated in tensile testing. Results are given in Table 3c and Fig. 6. In case of pure aluminum, a longer milling duration led to a reduction in TYS from 94MPa to 84MPa (-10%) while an increase in UTS from 116MPa to 126MPa (+9%) was observed. A reduction of TYS seemed to be very surprising since longer milling duration should lead to higher amount of deformation (generation of dislocations) in the Al-matrix and thus a work hardening effect. Hence, these experiments will be repeated in the future to figure out if that finding was a real effect that could maybe be related to softening by recovery or recrystallization due to significant heat generation during the milling process. For the 1.0%-SA-GNP composites an extension of milling duration resulted in increasing TYS from 81MPa to 95MPa (+17%) as well as increasing UTS from 112MPa to 141MPa (+26%). The fact that composite strengths increased could be related with improved SA-GNP dispersion in the composite (Fig. 3c) and hence a more effective strengthening effect of the graphene.

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Table 3. a) Tensile test results for composites containing SA-GNPs, processed via 6min mechanical mixing and 1min disc milling, extrusion with conic die (2α=130°) b) Effect of die angle for R=14:1 after 1min disc milling c) effect of disc milling duration (t_{DM}).

a)

GNP content	R	TYS [MPa]	UTS [MPa]	ε_{frac} [%]
0%	9:1	92±2	114±2	29±1
0.5%	9:1	85±2	110±2	28±1
1.0%	9:1	87±2	111±1	25±3
0%	14:1	94±2	116±1	28±2
0.5%	14:1	90±2	114±6	25±4
1.0%	14:1	90±2	116±2	27±1

b)

GNP content	die angle 2α	TYS [MPa]	UTS [MPa]	ε_{frac} [%]
0.5%	180°	87±3	113±2	32±3
0.5%	130°	90±2	114±6	25±4
1.0%	180°	89±2	115±2	22±8
1.0%	130°	90±2	116±2	27±1

c)

GNP content	t_{DM}	TYS [MPa]	UTS [MPa]	ε_{frac} [%]
0%	1min	94±2	116±1	28±1
0%	5min	84±2	126±1	29±4
1.0%	1min	81±1	112±1	23±1
1.0%	5min	95±1	141±1	17±3

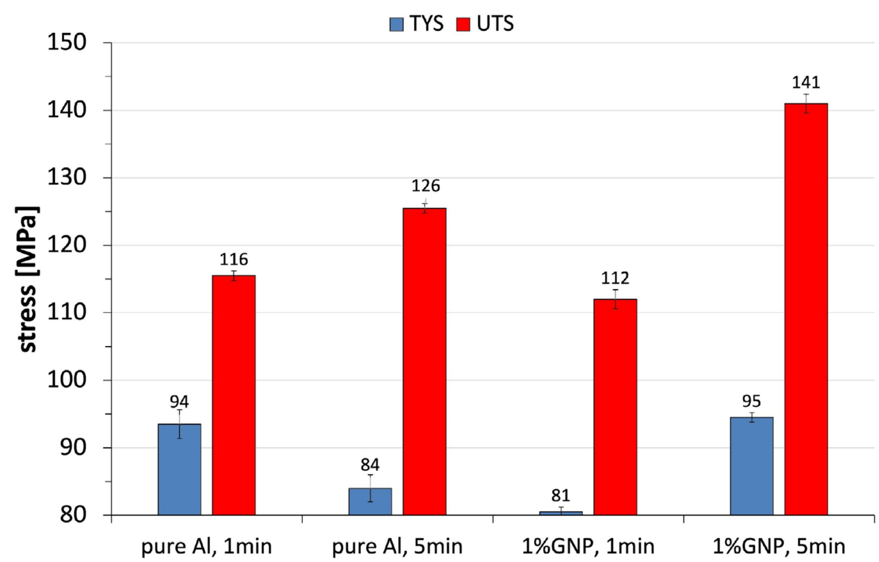

Fig. 6. Effect of disc milling duration on TYS and UTS of pure aluminum and 1%-SA-GNP composite rod materials.

When the strengths values of pure Al are compared to those of the 1% SA-GNP composite produced with same milling duration, it has to be noted that the addition of 1% GNP decreased TYS from 94MPa to 81MPa (-14%) after 1min of disc milling. UTS remained about constant and fracture strain decreased from 28% to 23%. Microstructural analysis with SEM will be carried out on the extruded rod material soon in order to investigate if maybe GNP agglomerations have formed locally for the relatively high graphene content of 1% and if those could be the reason for the detrimental YS. However, after a milling duration of 5min the addition of 1% SA-GNPs resulted in increased TYS from 84MPa to 95MPa (+13%) and UTS from 126MPa to 141MPa (+12%). Again, this could be due to improved GNP dispersion in the aluminum matrix after the longer milling duration and thus, an improved strengthening effect of graphene.

Since the results for SA-GNP composites were not as good as expected an alternative graphene source produced by Alfa Aesar (AA) was applied for the second part of the study. The effects of increasing AA-GNP content on micro hardness and tensile properties were investigated, after applying powder processing via mechanical mixing only on the one hand and additional 5min disc milling on the other hand.

Fig. 7a presents the effect of AA-GNP content on micro hardness. In case of extruded composites produced from powders that were mechanically mixed only, increasing the AA-GNP content did not affect micro hardness as it remained about constant around 35HV. But powder processing via 5min of disc milling step, significantly increased hardness of extruded rods from 36HV to 43HV (+20%) in case of the 0.25% AA-GNP composite. The addition of a higher AA-GNP content of 1.0% increased micro hardness even further to 54HV, which means an increase of 49%. However, hardness decreased to values comparable to pure Al when AA-GNP content was 0.5%. This result could not yet be explained. If upcoming microstructure analysis with SEM should not show significant GNP agglomeration these tests will be repeated for verification.

In Fig. 7b the results of TYS and UTS are presented in dependence of GNP content and powder processing approach. Very similar to the prior micro hardness findings, composites where powders were processed via only mechanical mixing prior to extrusion, the TYS and UTS values did not change significantly with increasing AA-GNP content. TYS remained about constant in the range between 76MPa and 82MPa. UTS values varied only slightly between 105MPa to 113MPa. Fig. 7c presents the results of the measured fracture strains. For the composites no effect was observed when AA-GNP additions of 0.25% and 0.5% were applied as fracture strain remained about constant at 28%. But increasing AA-GNP content to 1.0% resulted in significant reduction to a fracture strain of 18%. As a reason for that decrease the formation of GNP agglomerates is assumed.

When the effect of AA-GNP content is studied for the approach including the 5min disc milling step, it can be noted that TYS increased from 79MPa (pure Al) to 92MPa (+16%) for the 0.25% composite. The UTS also increased from 112MPa to 142MPa (+27%). The increase in composite strength is supposed to be based on the improved AA-GNP dispersion that was noticed in Fig. 3e. Fracture strain of 5min disc milled samples were found to decrease with increasing AA-GNP content it was found to be reduced from 32% (pure Al) to 21% for 0.25% AA-GNP and to 26% for 0.5% AA-GNP. As previously noticed for micro hardness values, the material with an AA-GNP content of 0.5% also featured a low TYS of only 77MPa and a UTS of only 112MPa, which is comparable to pure aluminum. This value marks a significant drop when being compared to the 0.25% AA-GNP composite (92MPa). This again is a reason to repeat the tests for this condition. Unfortunately, the samples with the highest AA-GNP content of 1.0% could not be characterized by tensile testing since the extruded rod featured significantly cracking and it was impossible to manufacture samples for tensile testing. Hence, this condition will also be part of upcoming investigations again.

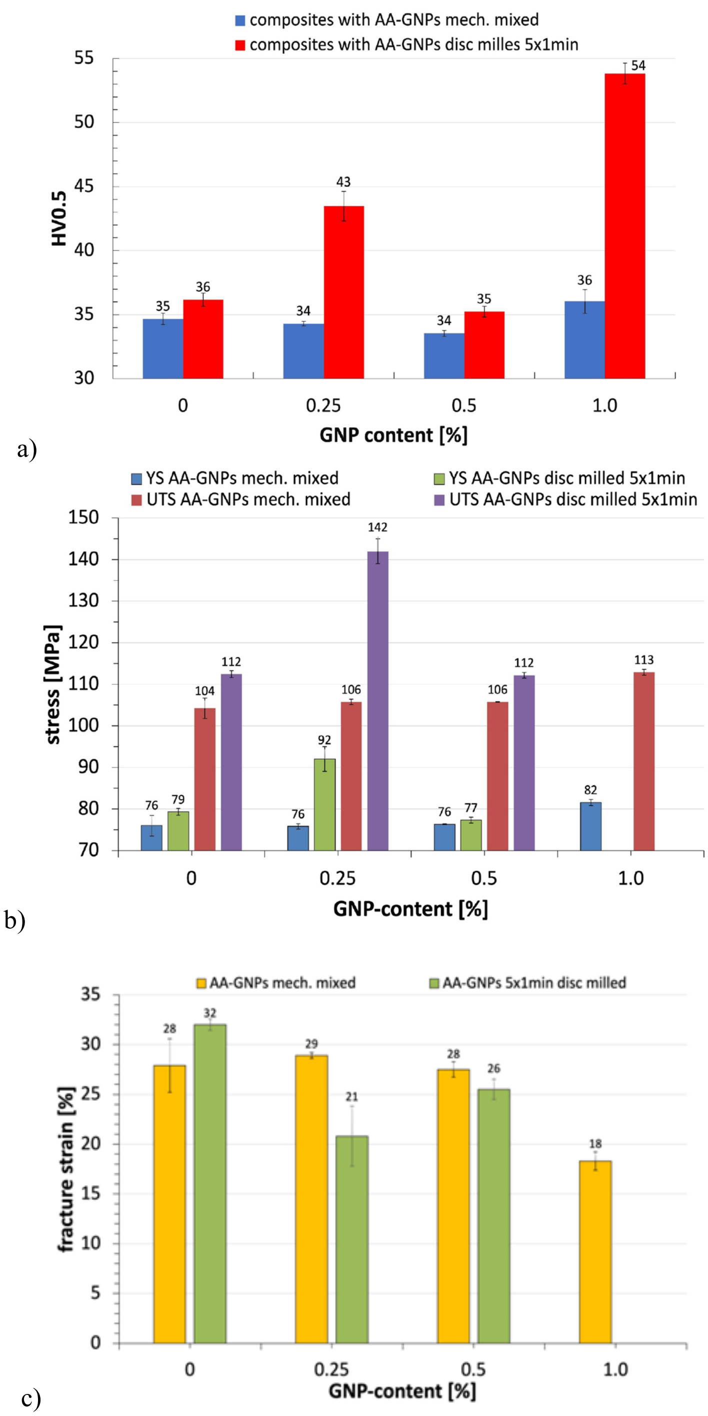

Fig. 7. Mechanical properties of extruded Al/GNP composite rods with different contents of AA-GNPs and initial powder processing via mechanical mixing only vs. additional 5min disc milling a) micro hardness b) TYS and UTS c) fracture strain.

Finally, when initial powder processing via mechanical mixing only is compared to the approach that featured the 5min disc milling, increased strength values were observed. For pure aluminum the increase in TYS was very low from 76MPa (mixed) to 79MPa (5min disc milled) and UTS from 104MPa to 112MPa (+8%). However, for the 0.25% AA-GNP composite 5min disc milling increased TYS from 76MPa to 92MPa (+21%) and UTS from 106MPa (mixed) to 142MPa (+34%). As previously assumed, the main reasons for increased strengthening are strain hardening of aluminum through plastic deformation during disc milling as well improved dispersion of GNPs in the aluminum matrix and thus an enhanced strengthening effect of the graphene additive.

Summary

The present study applied two different commercial graphene sources to investigate the disc milling process as a step in the powder metallurgical production chain for the manufacture of Al/GNP composites and the effects of process parameters on the mechanical properties. From the results the following conclusions can be drawn:

- When 1min of disc milling is applied, the addition of up to 1.0% of SA-GNPs did not significantly affect the mechanical properties of composites.
- Additionally, the application of either flat face die (2〈=180°) or conic die with 2〈=130°, as well as variation of extrusion ratio from 9:1, 14:1 and 31:1 did not have a significant effect on the mechanical composite properties.
- However, increasing the disc milling duration to 5min led to plate-like shaped Al-particles, a better exfoliation and dispersion of SA-GNPs and thus to increased micro hardness of up to 30%, increased TYS up to 17% and UTS up to 26%.
- Composites containing AA-GNPs improved in micro hardness by up to 49% (for 1% AA-GNPs) compared to pure Al, increased in TYS by up to 16% (for 0.25% AA-GNPs) and in UTS by up to 27% (0.25% AA-GNPs) when powders were disc milled for 5min.

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