

Letter to the Editor

Die wear during hot extrusion of *ex-situ* and *in-situ* aluminum composites

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Abstract

The study demonstrates a high-temperature wear of extrusion die during hot pressing of *in-situ* and *ex-situ* Al metal matrix composites (MMC). The *in-situ* Al-10.5vol.%AlN and *ex-situ* Al-10.5vol.%SiC with high Young's modulus are fabricated by powder metallurgy approach. The composite microstructure and the mechanical properties are characterized. Die wear during hot extrusion of MMC is quantified by the increase of radius of a worn edge as a function of the distance of extruded bars. *In-situ* Al-AlN exhibited a significantly lower abrasive effect in addition to higher mechanical properties compared to *ex-situ* Al-SiC.

Key words: aluminum, composite materials, extrusion, powder technology, wear

1. Introduction

Al-based MMC are attractive structural materials for use in aerospace, automotive and other industrial sectors [1, 2]. Al MMC possess good mechanical and physical properties, such as high specific strength and Young's modulus (E), a low coefficient of thermal expansion, and an increased wear resistance [3, 4]. The use of Al MMC with a high specific E leads to a significant weight reduction. Conventionally, stiffening phase in Al MMC with high E is introduced *ex-situ* typically as ceramic particulates, wherein SiC is the most popular because of its relatively high E and strength, good availability and low price [5, 6]. Traditional *ex-situ* Al MMC suffer from the problematic introduction of reinforcement, the damage of reinforcement and undesired interfacial reaction [3, 7]. Moreover, the minimum size of particulates, which can be homogeneously dispersed in the matrix, is limited to $\sim 1\text{--}10\ \mu\text{m}$. Consequently, coarse and abrasive ceramic particulates promote wear of production and post-processing tools [8, 9]. This becomes pronounced for thin-walled profiles fabricated by hot extrusion. Extrusion die wears quickly with a consequence of not meeting required dimensional tolerances. For this rea-

son, expensive extrusion dies have to be frequently replaced. It was shown that coating of extrusion dies suppresses a wear [10]. However, long term experience with industrial scale mass production of AlSi MMC extrudates proved that various coatings applied onto large and complex shape dies are inefficient in real operation [11]. This motivates the development of Al MMC fabricated *in-situ*, where disadvantages mentioned above can be avoided to a large extent. Numerous studies confirmed a benefit using *in-situ* Al MMC in terms of their beneficial mechanical and physical properties, creep and fatigue life performance, etc. [12, 13]. Though, information on the wear of production tools during fabrication of *in-situ* Al MMC at elevated temperatures lacks. For this purpose, in this study Al MMC stiffened with nano-scale AlN were fabricated *in-situ*. AlN is an attractive reinforcement for Al MMC as it features interesting properties similar to SiC, such as high E and strength, low density, low thermal expansion, and good wear resistance [14–16]. Furthermore, AlN is wetted by molten Al but does not react with it, where problems related to interfacial reactions, as with Al-SiC, are avoided. In the present work, the tribological behavior of extrusion die by *in-situ* Al-AlN at elevated tempera-

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Table 1. Mechanical properties of the composites. The breakthrough (P_{\max}) and minimal (P_{\min}) pressures determined during extrusion of composites

Composite	YS _{0.2} (MPa)	UTS (MPa)	ϵ (%)	E (GPa)	P_{\max} (MPa)	P_{\min} (MPa)
Al-AlN	210 ± 3	496 ± 5	2 ± 0.2	82.6 ± 1	637	420
Al-SiC 9 μm	114 ± 4	155 ± 4	11 ± 0.4	84.1 ± 0.9	732	555
Al-SiC 3 μm	125 ± 3	172 ± 5	11 ± 0.3	81.8 ± 1.3	532	358

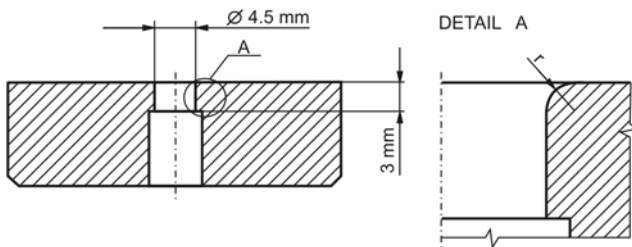


Fig. 1. (a) The drawing of the extrusion die and (b) the detail of worn edge on a die inlet.

ture is studied and attempted to quantify, and specific comparison to *ex-situ* Al-SiC with comparable E is made.

2. Experimental

To produce *in-situ* Al-AlN the Al (< 63 μm), Mg and Sn powders were used. Porous powder precursors were nitrided in flowing nitrogen in order to obtain 10.5 vol.% of AlN [17]. To produce *ex-situ* Al-10.5 vol.%SiC the Al (< 63 μm) and two different SiC powders ($d_{50} = 3$ and 9 μm) were used [18]. The Al-AlN and Al-SiC blanks were consolidated by direct extrusion (DE) realized at 450 °C into ϕ 4.5 mm diameter rods using a 180° nozzle die with an extrusion ratio of 44:1 (Fig. 1a). DE of one powder blank resulted in the extruded bar with the length of ~ 6 m. Three identical extrusion dies were manufactured from DIN X38CrMoV5-1 hot work tool steel (Böhler W300) with the resulting HV = 5737 ± 29 MPa and the average roughness R_a of 0.8 μm after heat treatment. Each die was used separately either for the extrusion of Al-AlN or Al-SiC powder blanks. After DE of one, three and five powder blanks the dies were cleaned from Al remnants by etching in NaOH solution. Cleaned dies were subjected to microstructural characterization. The extrusion pressure was monitored during DE. The density of MMC was measured using the Archimedes' principle. The microstructural characterization of MMC and dies was carried out using light microscopy (LM), transmission electron microscopy (TEM) and scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS). Changes of the edge radius (r) on the inlet of the

extrusion die (Fig. 1b) were determined from SEM images based on measurements taken at four different spots around inlet perimeter. Tensile tests were conducted using tensile bars with a gauge of ϕ 2–20 mm. The E of the extruded MMC was determined using dynamic mechanical analysis (DMA), a 3 point bending test using $2.5 \times 2.5 \times 55$ mm³ specimens.

3. Results and discussion

A small residual porosity of 0.3 and 0.1 % is confirmed for Al-AlN and Al-SiC, respectively. The microstructure of Al-AlN consists of two distinctive regions: (i) Al matrix and (ii) N rich bands (Figs. 2a,b). The elongated bands form as a result of shared nitrided shells on Al powder particles during DE. TEM reveals the composite character of the bands areas (i.e., fractured nitrided shells), where AlN crystals are embedded in Al matrix (Figs. 2c, d). The AlN particles have the size of tens or hundreds nm. *Ex-situ* Al-SiC is characterized by sharp edged SiC particles uniformly dispersed in Al (Figs. 2e,f). The SiC particles neither crack nor get distinctly arrayed or elongated along extrusion direction upon consolidation. Despite to a rather high shear deformation induced during DE (true strain ~ 3.8), the fine SiC particles in Al-SiC (3 μm) have tendency forming agglomerates. Even though a formation of SiC (3 μm) clusters is indistinctive, it can be expected that utilizing SiC particles finer than 3 μm would enhance formation of clusters.

Table 1 summarizes the mechanical properties of studied MMC. The E , as important mechanical property, is comparable for both Al-AlN and Al-SiC MMC. 10.5 vol.% of AlN and SiC yields ~ 20 % increase of E compared to the unreinforced Al matrix. Al-SiC 3 μm shows slightly lower E compared to Al-SiC 9 μm. This can be attributed to observed clustering of finer SiC 3 μm particles in Al matrix. Al-AlN shows the relatively high value of the 0.2 % strain offset yield stress (YS_{0.2}) of 210 MPa and ultimate tensile strength (UTS) of 496 MPa accompanied with relatively low total elongation (ϵ) of 2 %. This is attributed to *in-situ* nature of AlN, which contributes to good interfacial bonding and load transfer between Al matrix and AlN crystallites. Both Al-SiC exhibit comparable YS_{0.2}, UTS and ϵ . Compared to Al-AlN

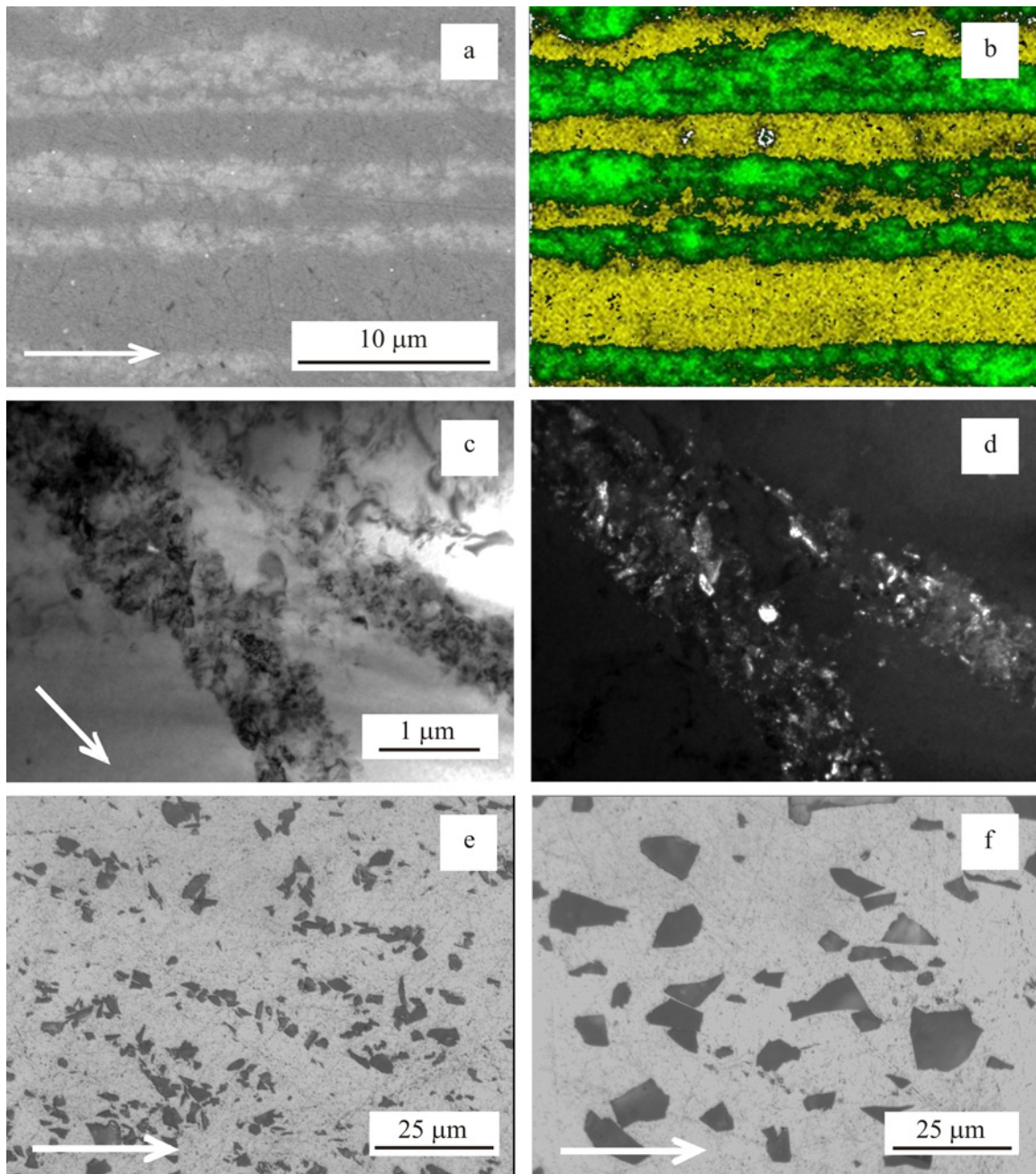


Fig. 2. Al-10.5 vol.%AlN: (a, b) SEM micrograph with the EDS elemental map of N (green) and Al (yellow), and (c, d) TEM micrograph with the corresponding dark field image showing nanoscale AlN crystals. LM micrographs of (e) Al-10.5 vol.%SiC 3 μm and (f) Al-10.5 vol.%SiC 9 μm . All micrographs show composites in the longitudinal direction. The arrows represent the extrusion direction.

the strength of Al-SiC deteriorates significantly. Conversely, the ε of Al-SiC increases to 11 %. An inferior ductility of Al-AlN is due to fractured nitrated shells at Al grain boundaries.

Figures 3a–d demonstrate wear of inlet edge on the extrusion dies after pressing of 30 m composite bars. It is assumed, that the inlet edges are perfectly sharp ($r = 0 \mu\text{m}$) on new as-fabricated dies (Fig. 3a). Apparently, extrusion dies made of hard tool steel get worn heavily during DE of both *ex-situ* Al-SiC (Figs. 3c,d),

where the dominant wear mechanism is abrasion with cracking of plastically deformed material between furrows. On the contrary, the inlet die edge after extrusion of Al-AlN is plastically deformed without signs of abrasion from AlN particles (Figs. 3b). These different mechanisms reflect the measured r values as the function of reinforcement size and shape. The wear rate, represented by an increase of the r over the distance of extruded bar, is nearly linear for Al-AlN and linear/parabolic for both Al-SiC (Fig. 4). Extrusion

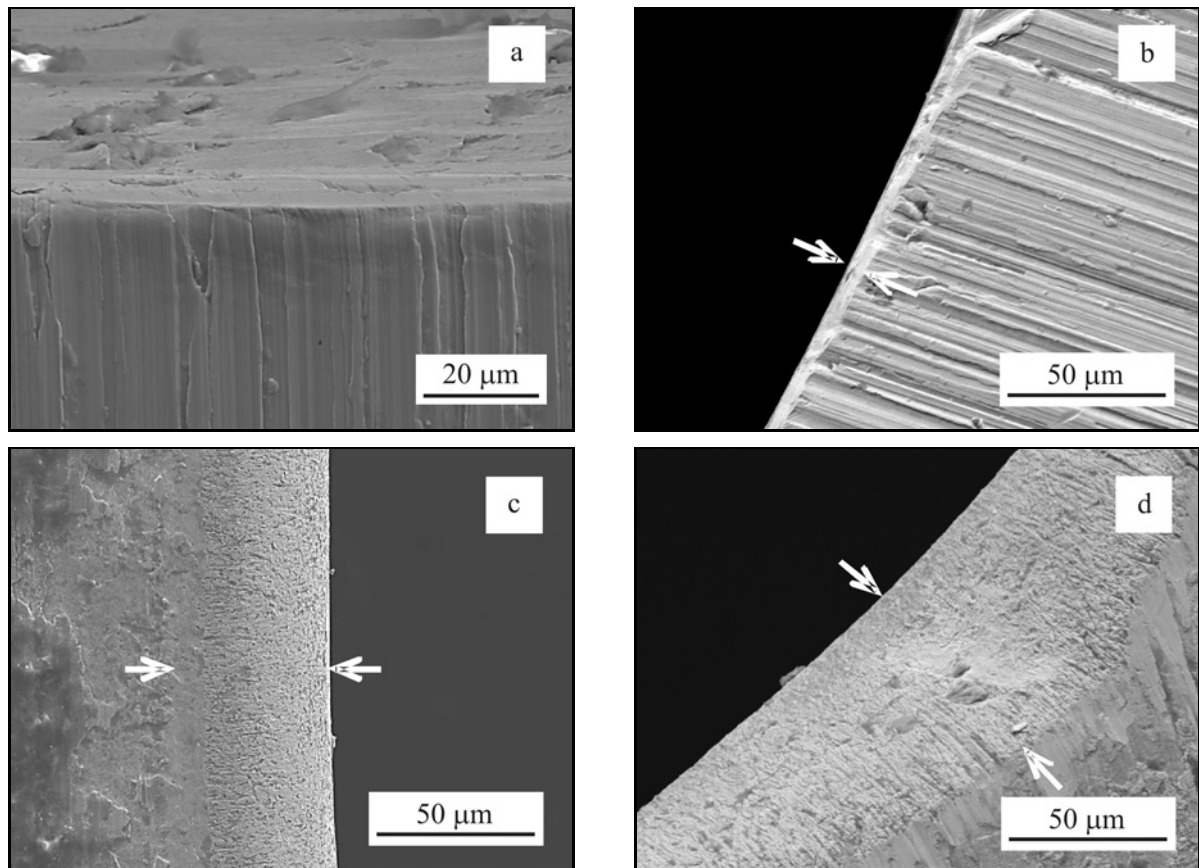


Fig. 3. SEM micrographs of the inlet edge of (a) as-fabricated extrusion die and the dies after extrusion of 30 m (b) Al-AlN, (c) Al-SiC 3 μm and (d) Al-SiC 9 μm composite bars. White arrows illustrate the radius of worn edge.

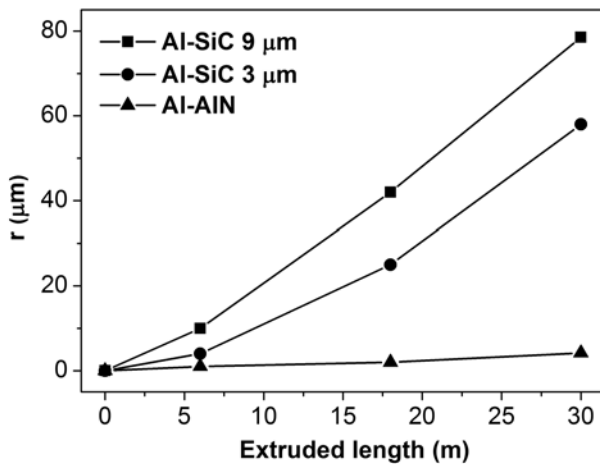


Fig. 4. Wear of extrusion dies represented by the radius (r) of a worn inlet edge as the function of the extruded length of Al-AlN and Al-SiC composite bars.

sion of 30 m long bar fabricated from Al-SiC 9 μm leads to a high value of an average $r = 79$ μm. Al-SiC with finer 3 μm SiC particles yields slightly reduced wear of die, which corresponds to $r = 55$ μm. It is apparent that after extrusion of rather small

quantity (30 m) of the highly abrasive Al-SiC extrusion die wears quickly and has to be changed in relatively short periods, in order to maintain required dimensional tolerances. It is expected that MMC which utilize finer SiC than 3 μm would result in less pronounced wear. Though, for homogenous dispersion of such fine SiC particles in Al matrix, high energy and time-consuming blending methods are required, which significantly increase costs and productivity. Al-AlN are significantly less abrasive and result only in $r = 4$ μm, what is being more than one order of magnitude less compared to Al-SiC. In order to exclude the effect of different contact pressure during extrusion of different MMC the extrusion pressures at the breakthrough event (P_{max}) and the point of extrusion termination (P_{min}) are monitored (Table 1). The reason for $P_{max} > P_{min}$ is a decrease of frictional forces as less and less material remains present in extrusion press container upon pressing. The extrusion pressures are highest for Al-SiC 9 μm and lowest for Al-SiC 3 μm. Despite the highest strength, the extrusion pressures of Al-AlN are between those determined for Al-SiC. Furthermore, it was shown that the extrusion pressure is not an important factor affecting a die wear [19, 20]. Based on these results, it can be concluded

that a wear of extrusion die in this study is mostly a function of size and morphology of reinforcing particle.

4. Conclusions

Wear of extrusion dies during hot compaction of high E *in-situ* Al-10.5 vol.%AlN and *ex-situ* Al-10.5 vol.%SiC was assessed. Al MMC with nano-scale AlN possessed superior mechanical strength and equal E compared to *ex-situ* Al-SiC. A die wear was quantified by the measuring of a worn radius after hot extrusion. Worn radius gradually increased with the length of extruded MMC bars (up to 30 m). Extrusion of Al-AlN accommodated more than one order of magnitude lower reduction of the worn radius. On the contrary, *ex-situ* Al MMC with micrometric SiC particles led to quick and heavy abrasion of extrusion dies.

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References

- [1] Ibrahim, I. A., Mohamed, F. A., Lavernia, E. J.: Journal of Materials Science, 26, 1991, p. 1115. [doi:10.1007/BF00544448](https://doi.org/10.1007/BF00544448)
- [2] Miracle, D. B.: Composites Science and Technology, 65, 2005, p. 2526. [doi:10.1016/j.compscitech.2005.05.027](https://doi.org/10.1016/j.compscitech.2005.05.027)
- [3] Torralba, J. M., da Costa, C. M., Velasco, F.: Journal of Materials Processing Technology, 133, 2003, p. 203. [doi:10.1016/S0924-0136\(02\)00234-0](https://doi.org/10.1016/S0924-0136(02)00234-0)
- [4] Rosso, M.: Journal of Materials Processing Technology, 175, 2006, p. 364. [doi:10.1016/j.jmatprotec.2005.04.038](https://doi.org/10.1016/j.jmatprotec.2005.04.038)
- [5] Sahin, Y., Acilar, M.: Composite: Part A, 34, 2003, p. 709. [doi:10.1016/S1359-835X\(03\)00142-8](https://doi.org/10.1016/S1359-835X(03)00142-8)
- [6] Ahlatci, H., Candan, E., Cimenoglu, H.: Wear, 257, 2004, p. 625. [doi:10.1016/j.wear.2004.03.006](https://doi.org/10.1016/j.wear.2004.03.006)
- [7] Tan, M. J., Zhang, X.: Materials Science and Engineering A, 244, 1998, p. 80. [doi:10.1016/S0921-5093\(97\)00829-0](https://doi.org/10.1016/S0921-5093(97)00829-0)
- [8] Goswami, R. K., Sikand, R., Dhar, A., Grover, O. P., Jindal, U. C., Gupta, A. K.: Materials Science Technology, 15, 1999, p. 443. [doi:10.1179/026708399101505914](https://doi.org/10.1179/026708399101505914)
- [9] Surappa, M. K.: Journal of Materials Science Letters, 12, 1993, p. 1272. [doi:10.1007/BF00506334](https://doi.org/10.1007/BF00506334)
- [10] Cooke, K. E., Yang, S., Selcuk, C., Kennedy, A., Teer, D. G., Beale, D.: Surface and Coatings Technology, 188–189, 2004, p. 697. [doi:10.1016/j.surfcoat.2004.07.003](https://doi.org/10.1016/j.surfcoat.2004.07.003)
- [11] <http://www.sapagroup.com/en/sapa-profile-as/our-offer/aluminium-profiles/new-materials/>, 2017 (accessed 16.03.17)
- [12] Krizik, P., Balog, M., Nosko, M., Riglos, M. V. C., Dvorak, J., Bajana, O.: Materials Science and Engineering A, 657, 2016, p. 6. [doi:10.1016/j.msea.2016.01.047](https://doi.org/10.1016/j.msea.2016.01.047)
- [13] Selcuk, C., Kennedy, A. R.: Materials Letters, 60, 2006, p. 3364. [doi:10.1016/j.matlet.2006.03.021](https://doi.org/10.1016/j.matlet.2006.03.021)
- [14] Balog, M., Krizik, P., Yan, M., Simancik, F., Schaffer, G. B., Qian, M.: Materials Science and Engineering A, 588, 2013, p. 181. [doi:10.1016/j.msea.2013.09.027](https://doi.org/10.1016/j.msea.2013.09.027)
- [15] Kondoh, K., Takeda, Y.: Powder Metallurgy, 43, 2000, p. 69. [doi:10.1179/pom.2000.43.1.69](https://doi.org/10.1179/pom.2000.43.1.69)
- [16] Yu, P., Balog, M., Yan, M., Schaffer, G. B., Qian, M.: Journal of Material Research, 26, 2011, p. 1719. [doi:10.1557/jmr.2011.143](https://doi.org/10.1557/jmr.2011.143)
- [17] Sercombe, T. B., Schaffer, G. B.: Acta Materialia, 52, 2004, p. 3019. [doi:10.1016/j.actamat.2004.03.004](https://doi.org/10.1016/j.actamat.2004.03.004)
- [18] Krizik, P., Balog, M., Matko, I., Svec Sr., P., Cavojsky, M., Simancik, F.: Journal of Composite Materials, 50, 2016, p. 99. [doi:10.1177/0021998315571028](https://doi.org/10.1177/0021998315571028)
- [19] Karamis, M. B., Nair, F.: Wear, 265, 2008, p. 1741. [doi:10.1016/j.wear.2008.04.044](https://doi.org/10.1016/j.wear.2008.04.044)
- [20] Nair, F., Karamis, M. B.: Tribology International, 43, 2010, p. 347. [doi:10.1016/j.triboint.2009.06.018](https://doi.org/10.1016/j.triboint.2009.06.018)