

A Comparative Study of Microstructure, Mechanical and Tribology Properties of Cast *in-situ* Particulate: Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) Composites

Abdulhaqq A. Hamid* S.C. Jain** P.K. Ghosh*** Subrata Ray***
abdulhaqqhamid@uomosul.edu.iq

* Mechanical Engineering Department, Collage of Engineering, University of Mosul, Mosul, Iraq

**Mechanical & Industrial Engineering Department, Indian Institute of Technology-Roorkee, Roorkee (Uttaranchal)-247 667 - India

***Metallurgical & Materials Engineering Department, Indian Institute of Technology-Roorkee, Roorkee (Uttaranchal)-247 667 - India

Received: 4/1/2022

Accepted: 20/2/2022

ABSTRACT

In-situ composites are a class of composite materials in which the reinforcing phases (such as Al₂O₃, TiB₂, TiC, etc.) are generated within the matrix material by some chemical reaction during the composite processing. This research paper compares the microstructure, mechanical and tribological properties of the resulting cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) composites. It is generally observed that intermetallic phase Mn(Al_{1-x}Fe_x)₆ in the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite is relatively finer in size and is sometimes blocky in type compared to Ti(Al_{1-x}Fe_x)₃ formed in cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite. This has been attributed to a difference in heterogeneous nucleation behavior of the alumina substrates during the phases of intermetallic formation. Superior mechanical properties, represented by ultimate tensile stress, yield stress and percentage elongation, are obtained in the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite and compared to those obtained in cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite. It is observed that the wear rate in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite is considerably lower compared to that of the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite, particularly at higher normal load of 39.2 N, in spite of a relatively higher porosity content and slightly lower hardness in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite.

Keywords: *In-Situ* Composites; Microstructure; Mechanical Properties; Dry Sliding

This is an open access article under the CC BY 4.0 license (<http://creativecommons.org/licenses/by/4.0/>).
<https://rengj.mosuljournals.com>

1. INTRODUCTION

Metal Matrix Composite (MMCs) can be fabricated via both the *ex-situ* and *in-situ* processing routes. The *in-situ* processing routes are often favoured for making MMCs, as these routes over-come the technical challenges encountered in the *ex-situ* processing routes. The technical challenges include non-uniform distribution of particles, interphase formation and poor wetting of reinforcement with the matrix material [1]. Aluminium based tribological materials have received considerable attention due to their ability to reduce the weight of components made out of them, leading to significant impact on fuel economy in dynamic systems. A variety of nonmetallic particles are dispersed in different metal systems to develop *ex-situ* discontinuous metal-matrix particulate composites (DMMPCs). Most researchers of *ex-situ* DMMPCs have found

a considerable increase in wear resistance owing to the reinforcement particles [2-6]. Based on the experience in composite containing externally added reinforcing particles, it is expected that the composites containing *in-situ* generated reinforcing particles may lead to important consequences in wear resistance and strength. Commonly used *in-situ* aluminium based composites are processed by the reaction between metal oxide and Al-melt [7]. *In-situ* particle reinforced aluminum alloy-based composites have been developed by solidification of slurry obtained by dispersion of externally added oxide particles (MnO₂ / TiO₂) in molten aluminum at a given processing temperature of 730 °C. The oxides have been chosen so that during processing, alumina (Al₂O₃) is formed by reaction of these oxide particles with molten aluminum. At the same time, the chemical reaction also releases alloying

elements like manganese / titanium into the molten aluminum, thus enhancing the value of the product. Some of the released alloying elements form a solid-solution with aluminum, while the remaining part, if there was any, reacts with aluminum to form intermetallic phases.

The present work is intended to investigate the characterizations, mechanical properties and wear and friction in cast *in-situ* Al(Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) composites. One of the most interesting observations of this system is the matrix strengthening achieved by alloying the composites with manganese / titanium when MnO₂ / TiO₂ particles are reduced by molten aluminium during processing. The research conducted here is expected to lead to an understanding of a situation where progressive matrix strengthening both by alloying and by the generation of hard particles during processing determine the overall mechanical and tribological behaviour.

2. EXPERIMENTAL PROCEDURE

Aluminium alloy-based composites containing *in-situ* generated alumina particles have been synthesized by stirring MnO₂ / TiO₂ particles into molten aluminium alloy followed by the addition of small amounts of surface active element of magnesium (Mg), according to procedures detailed in [8,9,10]. Both *in-situ* composites materials have been tested for mechanical properties and wear and friction. Dry sliding wear tests have been carried out by using a pin-on-disc machine. Different loads of 9.8, 19.6, 29.4 and 39.2 N have been applied on the pin, to the normal and the sliding contact, during wear test of each *in-situ* composite. The track radius has been kept constant at 50 mm and the linear speed has been maintained at 1.05 m/s, according to procedures detailed elsewhere in [10,11,12].

3. RESULTS AND DISCUSSIONS

Figure 1 shows a typical unetched SEM microstructure of cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) composites containing intermetallic phases, reinforcing particles, and a small number of pores. It is generally observed that intermetallic phase Mn(Al_{1-x}Fe_x)₆ in the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite is relatively finer and is sometimes blocky compared to Ti(Al_{1-x}Fe_x)₃ formed in cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite. This has been attributed to a difference in heterogeneous nucleation behavior of the alumina substrates during the formation of intermetallic phases. In the case of cast *in-situ*

Al(Mg,Ti)-Al₂O₃(TiO₂) composite, the intermetallic phases form due to the release of titanium into the matrix alloy due to chemical reduction of TiO₂ particles by molten aluminium. The intermetallic phases display both blocky (with an average size of about 5 μm) and platelike (with an aspect ratio of about four) shapes. No significant difference in the size and distribution of pores is observed in the microstructure of the different cast *in-situ* composites. However, there are large pores around clusters of particles observed at the top of the cast ingot.

Figure 2 shows a comparison of average Brinell hardness of cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite containing 2.726 wt% reinforcing particles and 1.973 vol% porosity, and cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite containing 2.904 wt% reinforcing particles and 1.282 vol% porosity.

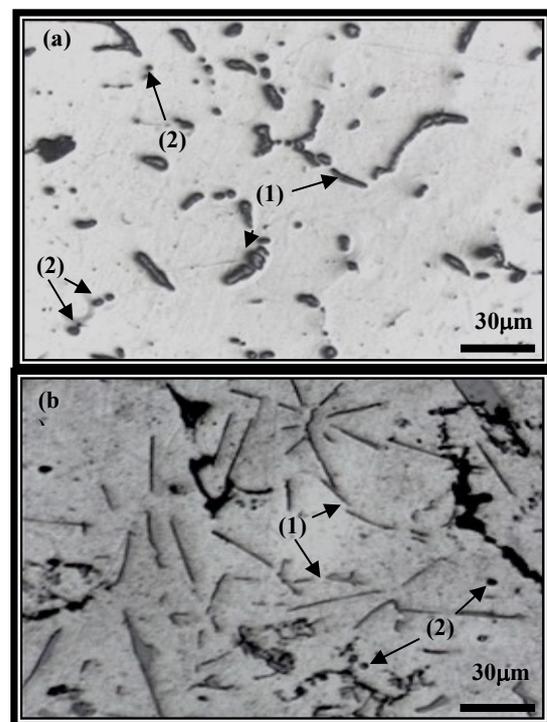


Figure 1. Unetched optical microstructure of cast *in-situ* composite (a) cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite; the intermetallic phase Mn(Al_{1-x}Fe_x)₆ marked by (1) and the reinforcing particles marked by (2) and (b) cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite; the intermetallic phase Ti(Al_{1-x}Fe_x)₃ marked by (1) and the reinforcing particles marked by (2).

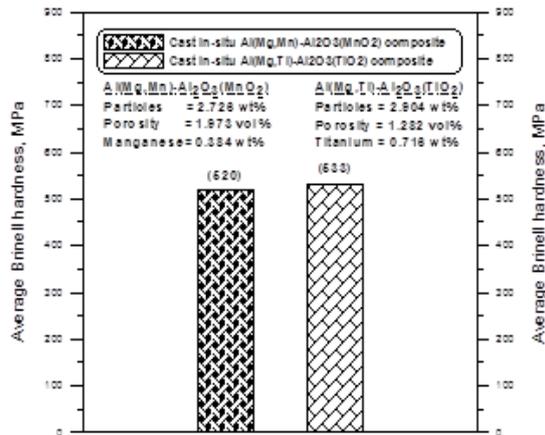


Figure 2. Comparison of Brinell hardness for cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite containing 2.726 wt% reinforcing particle and 1.937 vol% porosity, and cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite containing 2.904 wt% reinforcing particle and 1.282 vol% porosity.

The hardness in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite is slightly lower than that measured in the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite by about 3%. The porosity content of the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite is lower than that of the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite. Thus, the reduced hardness of the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite as compared to that of cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite could be attributed mainly to the relatively higher porosity.

Generally, the improvement in the mechanical properties resulted from the solid solution strengthening of Mn / Ti solute and fine particulate strengthening of Alumina [7,10]. If one compares the mechanical properties in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composites and those observed in cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composites on the basis of nearly similar particle contents and porosities, it is evident that the mechanical properties show significant differences in both of the systems. In the context of tensile properties, there are relatively higher tensile properties (ultimate tensile stress, yield stress, and percentage elongation) in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite as shown in Fig. 3 than those observed in the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite, as illustrated in the same figure, in spite of a relatively lower amount of reinforcing particles and higher porosity content in the former cast *in-situ* composite. Microstructural examination reveals that the precipitates of intermetallic phases is considerably finer in the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite

than in the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite. It is also observed that titanium bearing intermetallic phases are irregular, elongated, and sharp edged in the microstructure of the latter cast *in-situ* composites which are consequently acting as sites for stress concentration leading to relatively lower strength. The interfacial bonding between the matrix and the reinforcing particles may also be stronger in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite.

Figure 4(a) and (b) shows the variation of cumulative volume loss with sliding distance at normal loads of 9.8, 19.6, 29.4 and 39.2 N, and a fixed sliding speed of 1.05 m/s for the two cast *in-situ* composites. Figure 5 shows the variation in wear rate of these two cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) composites with normal load. It is observed that the cumulative volume loss and wear rate in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite are considerably lower, as shown in Fig. 4(a) and Fig. 5, than those in the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite, as illustrated in Fig. 4(b) and Fig. 5, particularly at higher normal load of 39.2 N, in spite of a relatively higher porosity content and a slightly lower hardness in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite. However, the comparison of results presented above clearly reveals the potential of both cast *in-situ* composites Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂). In the end, there is an urgent need for a better foundry practice for solidification processing of cast *in-situ* composites since cast *in-situ* composites containing relatively non-wettable particles of alumina have a natural tendency to form stable pore-particle combination which increases the overall porosity of the cast *in-situ* composites.

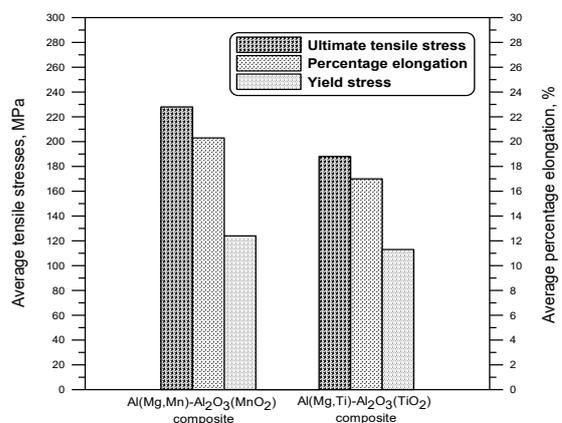


Figure 3. Comparison of tensile properties for cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) composites.

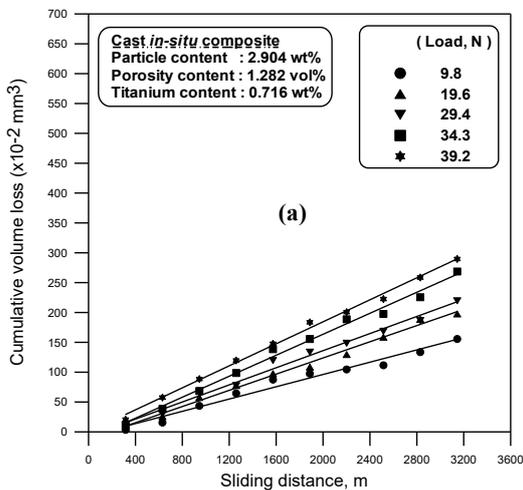
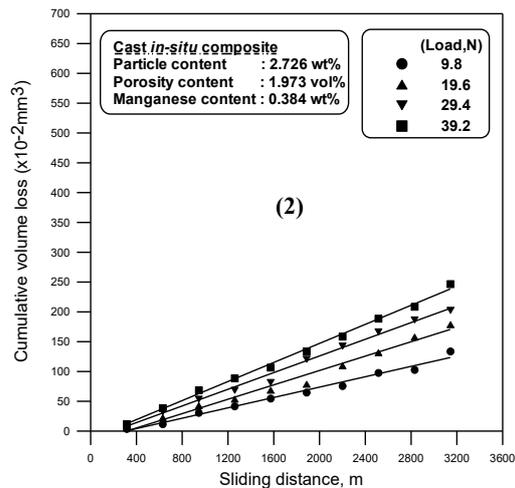


Figure 4. The variation of cumulative volume loss with sliding distance for (a) cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite and (b) cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite.

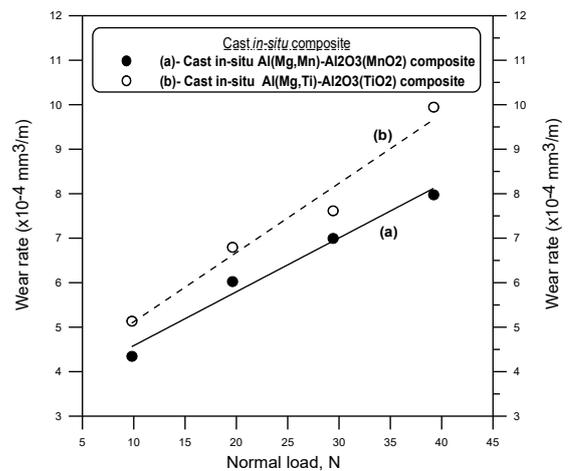


Figure 5. The variation of wear rate with normal load for cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) composites.

4. CONCLUSIONS

Based on the results presented above, the potential of both cast *in-situ* composites Al(Mg,Mn)-Al₂O₃(MnO₂) and Al(Mg,Ti)-Al₂O₃(TiO₂) is evident. A better foundry practice for solidification processing of cast *in-situ* composites is highly required since cast *in-situ* composites containing relatively non-wettable particles of alumina have a natural tendency to form stable pore-particle combination that increases the overall porosity of the cast *in-situ* composites. The study also concludes the following:

1. The microstructural examination reveals that the precipitates of intermetallic phase are considerably finer in the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite than those in the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite. This is attributed to a difference in heterogeneous nucleation behavior of the alumina substrates during the formation of intermetallic phases.
2. The hardness in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite is slightly lower than that measured in the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite by about 3%.
3. The tensile properties (ultimate tensile stress, yield stress, and percentage elongation) in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite are relatively higher compared to those observed in the

cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite with a similar amount of reinforcing particles and similar porosity presumably because of finer intermetallic phases in the former coupled with irregular and elongated shapes with sharp edge of the intermetallic phase in the latter. The interfacial bonding between the matrix and the reinforcing particles may also be stronger in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite.

4. The cumulative volume loss and wear rate in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite are relatively lower compared to those of the cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite with similar reinforcing particles and porosity, particularly at higher normal load of 39.2 N, in spite of a slightly lower hardness in cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite. Thus, the reduced volume loss and wear rate of the cast *in-situ* Al(Mg,Mn)-Al₂O₃(MnO₂) composite compared to those of cast *in-situ* Al(Mg,Ti)-Al₂O₃(TiO₂) composite could be attributed to relatively superior tensile properties and better interfacial bonding between the *in-situ* formed reinforcing particles and the matrix.

REFERENCES:

- [1] A.S. Vivekananda, et al. "processing-Structure-Property correlations of in-situ Al/TiB₂ Composites Processed by aluminothermic reduction process", Sci.Eng.Compo. Mater., 25(5), 2018, pp869-879.
- [2] P. K. Rohatagi, Y. Liu, and S. Ray, "Friction and Wear of Metal-Matrix Composites", ASME, Handbook, ed., Scott, D.Henry, 1997, Vol. 18, pp. 802-811.
- [3] Y. B. Liu, S. C. Lim, S. Ray, and P. K. Rohatagi, Wear, 1992, Vol. 159, pp. 201-205.
- [4] P. K. Rohatagi, S. Ray, and Y. Liu, Inter. Mater. Revs., 1992, Vol. 37, pp. 129-148.
- [5] A. T. Alpas and J. Zhang, Met. and Mater. Trans. A, 1994, Vol. 25A, pp. 969-983.
- [6] Manish Roy, B. Venkataraman, V. V. Bhanuprasad, Y. R. Mahajan, and Sundaraaian, Metall. Trans. A, 1992, Vol. 23A, pp. 2833-2846.
- [7] S. L. Pramod, et al, "Aluminium – Based Cast in-situ Composites: A Review" JMEPEG, ASM International, Feb., 2015.
- [8] A. Abdulhaqq Hamid, P. K. Ghosh, S. C. Jain, and S. Ray, Met. and Mater. Trans. A, 2005, Vol. 36A, pp. 2211-2223.
- [9] A. Abdulhaqq Hamid, P. K. Ghosh, S. C. Jain, and S. Ray, Met. and Mater. Trans. A, 2006, Vol. 37A, pp. 469-480.S.
- [10] A. Abdulhaqq Hamid, Ph.D. "Thesis, Aluminim based *in-situ* composites-synthesis, mechanical and tribological properties", IIT-Roorkee, India, 2006.
- [11] A. Abdulhaqq Hamid, P. K. Ghosh, S. C. Jain and S. Ray, "Influence of Particle Content and Porosity on the Wear Behaviour of Cast In-Situ Al(Mn)-Al₂O₃(MnO₂) Composite", Wear, 2006, Issues No. 4-5, Vol. 260, pp. 368-378.
- [12] A. Abdulhaqq Hamid, P. K. Ghosh, S. C. Jain and S. Ray, "Influence of Particle Content and Porosity on the Wear Behaviour of Cast In-Situ Al(Ti)-Al₂O₃(TiO₂) Composite", Wear, 2008, Vol. 265, Issues 1-2, (25 June), pp. 14-26.

مقارنة بين البنية المجهرية، الخواص الميكانيكية والترايبولوجية للمترابكات المسبوكة والمعززة من الداخل بالحبيبات:

Al(Mg,Mn)-Al₂O₃(MnO₂) و Al(Mg,Ti)-Al₂O₃(TiO₂)

Subrata Ray***

P.K. Ghosh***

S.C. Jain**

عبد الحق عبد القادر الدباغ*

abdulhaqqhamid@uomosul.edu.iq

*جامعة الموصل - كلية الهندسة - قسم الهندسة الميكانيكية- موصل - العراق

** قسم الهندسة الميكانيكية والصناعية ، المعهد الهندي للتكنولوجيا - روركي ، روركي (أوتارانشال) - 247667 - الهند

*** قسم هندسة المعادن والمواد ، المعهد الهندي للتكنولوجيا - روركي ، روركي (أوتارانشال) - 247667 - الهند

الملخص

المترابكات المعززة من الداخل هي احد أصناف المواد المترابكة والتي يتكون فيها الطور المعزز من الداخل نتيجة تفاعل كيميائي اثناء عملية التصنيع. في البحث الحالي، تمت مقارنة البنية المجهرية، الخواص الميكانيكية والترايبولوجية للمترابكات المسبوكة والمعززة من الداخل بالحبيبات ($Al(Mg,Mn)-Al_2O_3(MnO_2)$) و $Al(Mg,Ti)-Al_2O_3(TiO_2)$. وبصورة عامة، تم ملاحظة بان الطور البيني المعدني $(Mn(Al_{1-x}Fe_x)_6)$ المتكون في المترابك المسبوك $(Al(Mg,Mn)-Al_2O_3(MnO_2))$ هو نسبيا اصغر بالحجم وعلى شكل كتل صغيرة مقارنة مع الطور البيني المعدني $(Ti(Al_{1-x}Fe_x)_3)$ المتكون في المترابك المسبوك $(Al(Mg,Ti)-Al_2O_3(TiO_2))$. ويمكن اعزاء السبب لذلك نتيجة اختلاف سلوك التكوين العشوائي للألومينا (Al_2O_3) المتكونة مع الطور البيني المعدني في المترابك المسبوك $(Al(Mg,Mn)-Al_2O_3(MnO_2))$. كما تبين من الدراسة الحالية بان الخواص الميكانيكية والترايبولوجية للمترابك المسبوك $(Al(Mg,Mn)-Al_2O_3(MnO_2))$ هي افضل من تلك للمترابك المسبوك $(Al(Mg,Ti)-Al_2O_3(TiO_2))$. تبين بان معدل البلى للمترابك المسبوك $(Al(Mg,Mn)-Al_2O_3(MnO_2))$ اقل من معدل البلى للمترابك المسبوك $(Al(Mg,Ti)-Al_2O_3(TiO_2))$ ، وخصوصا عند الحمل الكبير (39.2 N) على الرغم من كثرة الفجوات وانخفاض الصلادة للمترابك المسبوك $(Al(Mg,Mn)-Al_2O_3(MnO_2))$.

الكلمات الداله :

المترابكات المعززة من الداخل، البنية المجهرية، الخواص الميكانيكية، الانزلاق الجاف.