NanoWorld Journal

https://doi.org/10.17756/nwj.2023-s3-131

Research Article

Open Access

Investigating Corrosion Response and Mechanical Performance of Al/B_4C and Al/TiO_2 Metal Matrix Nanocomposites Synthesized by Powder Metallurgy

Ashvin B Amale[1*], K.P. Dhanabalakrishnan[2], P. Muraleedharan[3], Prashant Kumar Gangwar[4], P. Satishkumar[5], Nashmi H. Alrasheedi[6], K. Karpagavalli[7] and J. Sandhiya Jenifer[7]

[1]*Department of Mechanical Engineering, Sri Eshwar College of Engineering, Coimbatore, Tamil Nadu, India*
[2]*Department of Aeronautical Engineering, Hindusthan Institute of Technology, Coimbatore, Tamil Nadu, India*
[3]*Department of Mechanical Engineering, Karpagam College of Engineering, Coimbatore, Tamil Nadu, India*
[4]*Department of Construction Technology and Management, Woldia Institute of Technology, Woldia University, Woldia Town, Ethiopia*
[5]*Department of Mechanical Engineering, Rathinam Technical Campus, Coimbatore, Tamil Nadu, India*
[6]*Department of Mechanical and Industrial Engineering, College of Engineering, Imam Mohammad Ibn Saud Islamic University, Riyadh, Saudi Arabia*
[7]*Department of Physics, Rathinam Technical Campus, Coimbatore, Tamil Nadu, India*

[*]Correspondence to:
Ashvin B Amale
Department of Mechanical Engineering,
Sri Eshwar College of Engineering,
Coimbatore, Tamil Nadu, India.
E-mail: abamale@ycce.edu

Received: July 31, 2023
Accepted: October 30, 2023
Published: November 01, 2023

Citation: Amale AB, Dhanabalakrishnan KP, Muraleedharan P, Gangwar PK, Satishkumar P, et al. 2023. Investigating Corrosion Response and Mechanical Performance of Al/B_4C and Al/TiO_2 Metal Matrix Nanocomposites Synthesized by Powder Metallurgy. *NanoWorld J* 9(S3): S739-S744.

Published by United Scientific Group

Abstract

In this study, researchers used powder metallurgy to create a variety of aluminum/boron carbide (Al/B_4C) and aluminum/titanium dioxide (Al/TiO_2) metal matrix nanocomposites (MMNCs). The manufactured MMNCs were tested to determine their mechanical qualities. In addition, the MMNCs' corrosion behavior in sodium chloride (NaCl) aqueous solution was studied at varying concentrations of NaCl, temperatures of the solution, and times of exposure. Taguchi's method for designing experiments was used to create the corrosion tests. Calculations of MMNC corrosion rates (CR) were made in relation to test parameters. Al/TiO_2 and Al/B_4C MMNCs are shown to have significantly higher hardness than pure Al matrices. The toughness of MMNCs grows as their nanoparticle volume fraction and/or size grows. Corrosion resistance of Al/B_4C and Al/TiO_2 MMNCs was significantly higher than that of pure Al in NaCl aqueous solution. Corrosion of the Al/TiO_2 MMNCs was less rapid than that of the Al/B_4C MMNCs. The CR of MMNCs was shown to be primarily influenced by the exposure time and the temperature of the NaCl solution, according to physical and statistical studies.

Keywords

Metal matrix nanocomposites, Nanocomposites, Corrosion, Powder metallurgy, Aluminium alloys

Introduction

Corrosion occurs when a material reacts with its environment, either chemically or electrochemically, leading to dissolution, leaching, and deterioration. Material structure, both internal and external, and environmental factors determine whether corrosion is localized or widespread. Corrosion happens everywhere with homogenous single-phase materials. Uneven corrosion can also be caused by the separation of reactive contaminants and phases at grain boundaries [1].

Due to their low weight, high specific strength, and high stiffness, metal matrix composites (MMCs) find widespread application in nautical engineering. They are also resistant to wear and friction at both low and high temperatures [2, 3]. These materials need to be corrosion-resistant so that they can be used in salt water. Reinforcements such as particles, fibers, and whiskers (short fibers) are scattered throughout the metal matrix of an MMC, which combines two or more components with differing corrosion potentials and corrosion properties [4].

Corrosion in the matrix could be hastened by electrochemical, chemical, or physical interactions with reinforcing particles, fibers, or whiskers. The reinforcement and matrix may galvanically interact, which might speed up corrosion. When particles and a matrix are joined together in a composite, preferred corrosion can seriously reduce the interfacial area. This can cause MMCs to corrode more quickly than their corresponding monolithic matrix alloys [5, 6]. The matrix's inhomogeneities can be addressed in a number of ways, such as by crevice assault at metal/reinforcement interfaces or through favored localized attack on inhomogeneities in the matrix's composition and structure. Corrosion in corrosive conditions, especially when combined with stresses, reduces the load-bearing capacity of MMCs, potentially leading to catastrophic failures [7].

While research on MMCs' physical and mechanical qualities is extensive, corrosion behavior is not noted [8]. Traditional MMC development focused on matching mechanical qualities to specific applications, while corrosion studies were an afterthought.

The modulus of elasticity and other physical and mechanical parameters of composites can be estimated using models and equations that account for the reinforcements and the matrix, the geometry, chemistry, and volume fractions of reinforcements [9, 10]. Because processing has such a significant impact on the corrosion behavior of MMCs, there are currently no prediction formulas available for this phenomenon. MMNCs have lately acquired appeal in a variety of scenarios because of their superior attributes compared to those of conventional microparticle reinforced MMCs. Incorporating ceramic nanoparticles into MMCs is thought to significantly improve their characteristics, even at a modest volume percentage [11]. Academics and businesses alike are enthusiastic about the possibilities presented by these MMNCs. Corrosion resistance in metals like Al and its alloys may be increased by incorporating ceramic nanoparticles into the matrix. However, there is a paucity of efforts examining the corrosion behavior of MMNCs, just as there is with regular MMCs [12]. The corrosion behavior of MMNCs needs further study, thus researchers must conduct further experiments.

The current research aims to learn more about Al/B_4C and Al/TiO_2 MMNCs' mechanical and corrosion properties. Using the standard powder metallurgy method, many Al MMNCs with varying amounts of B_4C and TiO_2 nanoparticles of varying sizes were produced. Taguchi's approach of experimental design was used to plan the corrosion testing. The CR was calculated using the weight loss technique. Several corrosion tests were conducted at temperatures ranging from 0 to 75 °C. Test periods lasted anywhere from 120 to 240 h of exposure. This study examined the influence of different material factors, such as size, type of reinforcement, and volume fraction, as well as corrosion test conditions, including solution temperature, concentration of NaCl solution, and exposure time, on the behavior of corrosion of the MMNCs in comparison to the pure Al matrix. Correlations were also developed mathematically between these factors and the pace at which MMNCs corroded in NaCl. Such correlations allow for the prediction of the MMNC CR.

Experimental Procedure

Materials

The matrix material in this investigation was an Al powder with a minimum purity of 99.7% that was obtained from commercial sources. The micron size of the Al powder ranged from 10 to 100. Reinforcing agents included both B_4C and TiO_2 ceramic nanoparticles. The typical diameters of B_4C and TiO_2 nanoparticles are 210 and 70 nm, respectively.

Fabrication of MMNCs

The conventional powder metallurgy method was utilized to produce a large number of MMNCs containing up to 6 vol.% B_4C and TiO_2 nanoparticles: Mechanical blending was used to create a consistent mixture of Al powder and nanoparticles with 0.6 - 1.8 wt.% paraffin lubricating wax, which was then placed in containers for later use. The powders consisting of Al/nanoparticles were subjected to cold compaction using a die made of tool steel, as depicted in figure 1. In a 400 kN hydraulic press, the particles were then compacted. The compaction process was carried out at a pressure of 500 MPa. The MMNCs were then sintered at 600 °C for 100 min following the cold compaction stage. Argon was used as an inert gas during the sintering process. The MMNCs were heated in an extruder after being sintered [13]. At 500 °C, MMNC was extruded into billets. The upper cylinder was wrapped in nickel-chromium coils for heating purposes. The area reduced by the extrusion was halved. Cylindrical in shape, the final MMNC samples measured in at ~100 mm in length and 5 mm in diameter.

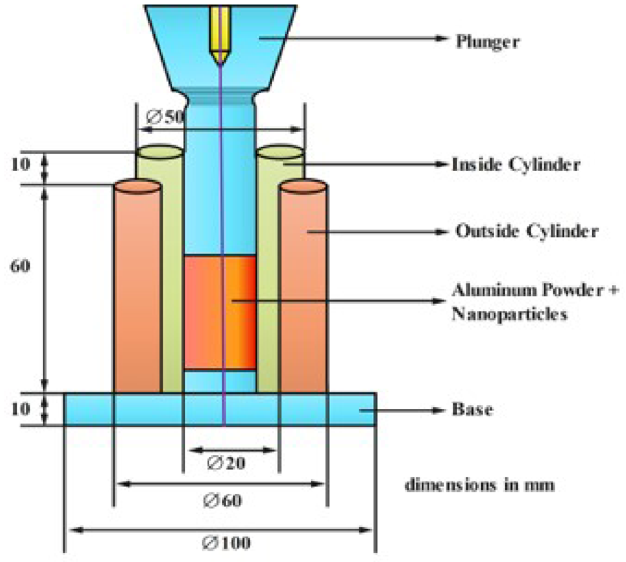

Figure 1: Diagrammatic representation of a cold compaction die.

Porosity measurements of MMNCs

ASTM B962-08.13 specifies the use of the water displacement (Archimedean density) method to determine the bulk porosity of manufactured MMNCs.

Microhardness measurements

A Roll/Zwick microhardness tester was used to determine the hardness of the MMNC samples after they were cleaned. A Vickers indentation was used to deliver an indentation force of 25 g in accordance with ASTM B72191.14 [14]. Each sample had at least ten measurements taken of it, from which an average was calculated and presented.

Static immersion corrosion tests

Taguchi's design of experiments (DoE) methodology was used to develop static immersion corrosion testing. MiniTab commercial statistical software was used to conduct the DoE and analysis of variance. Al MMNCs' corrosion properties (CR) in NaCl aqueous solution were studied, and factors including nanoparticle type, volume fraction, size, solution concentration, solution temperature, and exposure duration were analyzed. Taguchi's mixed-level DoE method is chosen. For this analysis, we used a Taguchi orthogonal array with dimensions of L18.

The levels of the variables under investigation were maintained at a constant value, as presented in table 1. In this context, A represents the size of the nanoparticles in nanometers, B denotes the volume fraction of nanoparticles in percentage, C signifies the concentration of NaCl in weight percent, D represents the temperature of the solution in degrees Celsius, and E denotes the time of exposure in hours. Table 2 presents the experimental design for conducting an immersion corrosion test in an aqueous solution of NaCl. Circular samples were immersed in an aqueous solution of NaCl for the purpose of conducting corrosion tests [15]. The specimens were immersed in the solution using a plastic thread as a precautionary measure to prevent crevice corrosion. The ASTM-G31 standard's protocols were followed throughout the corrosion testing, and the results were assessed by calculating the percentage of weight loss. The samples were dried after being powdered, polished, washed in deionized water, rinsed in methanol, and then submerged in NaCl solution. Many hours were spent allowing the samples to soak in the test solution. The samples were then washed in 50% nitric acid for 3 min, dried, and weighed using a digital scale accurate to the nearest 0.1 mg, all in accordance with ASTM G1-90. The CR was calculated using the mass lost to corrosion using equation 1 [16, 17].

$$CR = \frac{K.W}{A.D.T} \tag{1}$$

In this context, 'CR' represents the corrosion rate in millimeters per year, 'T' denotes the duration of exposure in hours up to the next 0.01 h, 'K' stands for a constant value of 8.76 x 10^4, 'A' represents the surface area in square centimeters during the 0.01 h interval, 'D' signifies the density of the material in grammes per cubic centimeter, and 'W' indicates the weight loss in milligrams.

There were also corrosion experiments conducted at 50 °C and 75 °C, in addition to those conducted at ambient temperature (accelerated corrosion tests). An electric furnace was used to heat a solution of water and NaCl to an exact temperature (± 2 °C) in order to conduct the accelerated test [18]. The samples were placed in the heated solution, and the vessel was covered with a glass lid to prevent loss of moisture. Several hours were spent soaking the specimens in the test solution. After being exposed, the samples were processed through a series of cleaning and drying steps before being weighed on a 0.1 mg resolution digital balance.

Table 1: Analyzed the factors and levels used in NaCl aqueous solution immersion corrosion tests.

Factors	Symbol	Units	Level			Interval
			1	**2**	**3**	
A - Nanoparticles size	A	nm	70	140	210	70
B - Volume fraction	B	vol.%	2	4	6	2
C - NaCl concentration	C	wt.%	2	4	6	2
D - Solution temperature	D	°C	30	60	90	30
E - Exposure time	E	h	140	200	260	60

Note: The parameter's minimum, average, and maximum values are denoted by levels 1, 2, and 3, respectively.

Table 2: Corrosion test experiments in a matrix.

Run No.	Factor level				
	A	**B**	**C**	**D**	**E**
1	1	1	1	1	1
2	1	1	2	2	2
3	1	1	3	3	3
4	1	2	1	1	2
5	1	2	2	2	3
6	1	2	3	3	1
7	1	3	1	2	1
8	1	3	2	3	2
9	1	3	3	1	3
10	2	1	1	3	3
11	2	1	2	1	1
12	2	1	3	2	2
13	2	2	1	2	3
14	2	2	2	3	1
15	2	2	3	1	2
16	2	3	1	3	2
17	2	3	2	1	3
18	2	3	3	2	1

Results and Discussion

Porosity of MMNCs

Extruded pure Al matrix, Al/TiO_2 MMNCs, and Al/B_4C MMNCs samples were all measured for porosity, and the findings are depicted in figure 2. The processing technology used in this study produced MMNCs with a bulk porosity of less than 4 vol.%, proving its near net shape forming capabilities. When compared to the Al/TiO_2 and Al/B_4C MMNCs, the pure Al matrix had the least amount of porosity. The results show that adding more TiO_2 and/or B_4C nanoparticles to the MMNCs slightly increases their porosity. Al/TiO_2 MMNCs reinforced with 70 nm nanoparticulates showed a somewhat greater porosity content compared to 210

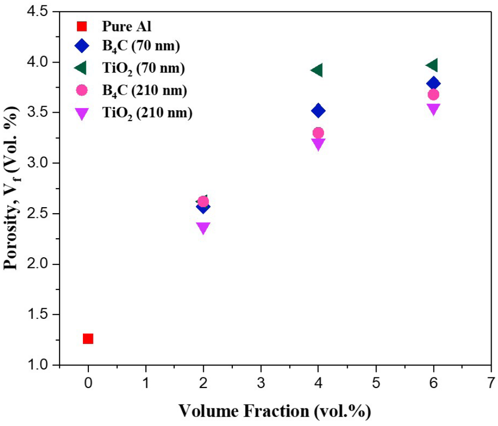

Figure 2: Difference of bulk porosity with volume fraction of MMNCs.

nm nanoparticulate-reinforced MMNCs. In contrast, the Al/B_4C MMNCs reinforced with 70 and 210 nm showed nearly the same porosity.

Microhardness of MMNCs

The microhardness of MMNCs varied with nanoparticle volume percentage as displayed in figure 3. The microhardness of Al/TiO_2 and Al/B_4C MMNCs was found to be greater than that of pure Al. Raising the percentage of nanoparticles in Al/TiO_2 and Al/B_4C nanocomposites raises their microhardness. It has also been discovered that by increasing the nanoparticle size from 70 to 210 nm while retaining the same volume fraction, the microhardness of Al/TiO_2 and Al/B_4C MMNCs can be enhanced. Microhardness was found to be higher in Al/B_4C MMNCs than in Al/TiO_2 MMNCs despite both types having the same volume percent and nanoparticulate size. Microhardness values were highest for the Al/B_4C MMNCs, which included 6 vol.% B_4C nanoparticulates of 210 nm.

Many researchers found that incorporating ceramic nanoparticles into MMNCs increased their toughness [19]. Microhardness in composites reinforced with B_4C nanoparticulates was found to be substantially higher than in pure AZ91D [20]. When comparing AZ91D/5SiC to magnesium AZ91D alloy, the average hardness is enhanced by ~75%. Magnesium based composites enhanced with B_4C nanoparticles showed increasing microhardness as B_4C nanoparticle content increased.

Behavior of MMNCs corrosion in a NaCl aqueous solution

CRs for the pure Al matrix and the MMNCs from all experiments conducted so far are depicted in figure 4. Corrosion tests were performed on a pure Al matrix, as shown in table 2. In comparison to the Al matrix, MMNCs have been proven to be more corrosion resistant. Al/TiO_2 MMNCs also showed enhanced corrosion resistance in comparison to Al/B_4C MMNC.

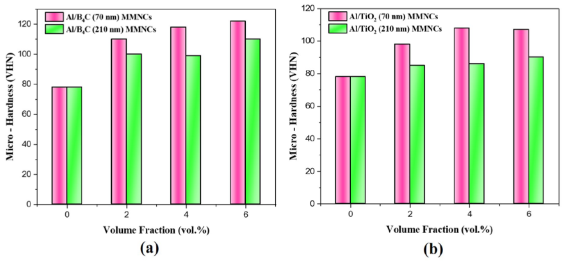

Figure 3: Microhardness evaluation of **(a)** Al/B_4C and **(b)** Al/TiO_2 MMNCs with volume fraction of nanoparticles of varying sizes.

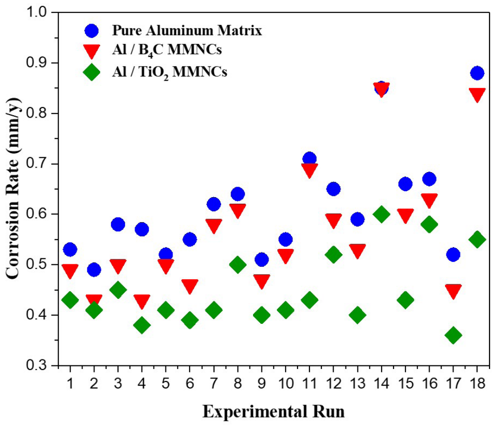

Figure 4: CR in a pure Al matrix and in MMNCs.

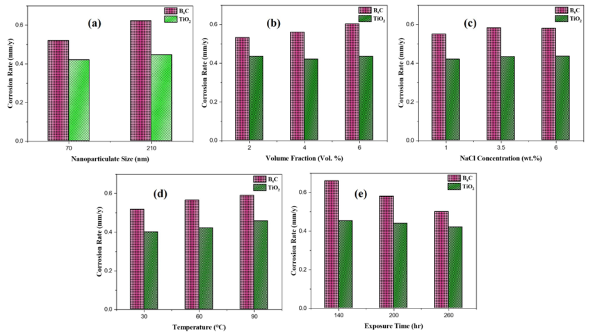

Figure 5: Variation of immersion corrosion test parameters on the CR of MMNCs.

Figure 5 displays the correlation between the parameters of a static immersion corrosion test and the MMNCs' CR. CR for both Al/TiO_2 and Al/B_4C MMNCs were shown to increase with increasing nanoparticle size from 70 to 210 nm (Figure 5). CRs of the MMNCs are also demonstrated to grow from 1 to 6% as the NaCl solution concentration is increased. Both Al/B_4C and Al/TiO_2 MMNCs exhibit this behavior. At higher temperatures, the NaCl aqueous solution corroded the MMNCs much more quickly. The CR is found to be linearly proportional to the temperature. Longer periods of exposure have been observed to slow the CR.

As shown in figure 5, raising the volume fraction of the TiO_2 nanoparticulates reduced the CR of Al/TiO_2 MMNCs. When the concentration of TiO_2 nanoparticles was doubled (from 4 to 6 vol.%), the CR doubled again. It was discovered that raising the B_4C nanoparticle volume fraction from 2 vol.% to 4 vol.% had no effect on the CR of Al/B_4C MMNCs. The

CR was decreased when the volume fraction of B_4C nanoparticles in the Al/B_4C MMNCs was raised from 2 to 6 vol.%.

The statistical analysis reveals that the CR of MMNCs is mostly affected by the temperature of the NaCl aqueous solution, whereas the period of immersion has a relatively minor influence on the CR.

Equation 2 and equation 3 describe the relationship between the CR of Al/TiO_2 and Al/B_4C MMNCs and the investigated corrosion testing variables [21].

$$CR_{TiO_2} = 0.175 + 0.000244S + 0.00048V + 0.00497C + 0.00114T - 0.000534D \quad (2)$$

$$CR_{SiC} = 0.367 + 0.000876S + 0.0163V + 0.00799C + 0.00127T - 0.00167D \quad (3)$$

Where 'S' is the nanoparticle size in nanometers, 'V' is the nanoparticle volume fraction in volume percent, 'T' is the NaCl solution temperature in degrees Celsius, 'C' is the NaCl solution concentration in weight percent, and 'D' is the exposure period in days (h).

The CR of the studied MMNCs (3) can be estimated using equation 2 and equation 3. The CR vs exposure time and solution temperature for Al/TiO_2 and Al/B_4C MMNCs are depicted in figure 6 and figure 7, respectively. These numbers show that as the temperature of the solution rises, so does the CR of the MMNCs. Additionally, as exposure time to the MMNCs increases, the CR reduces. Such findings have been experimentally stated before.

Authors [11] also showed that the dispersion of nanoparticles in an Al matrix increased the matrix's resistance to corrosion. Their work involved the fabrication of A356/TiO_2 MMNCs using rheocasting (the TiO_2 nanoparticle size was 500 nm). When comparing the A356 monolithic alloy to the A356/TiO_2 MMNCs, they found that the former had much higher corrosion rates in a 4% NaCl solution at room temperature. As an added bonus, they demonstrated that A356 alloy corrosion resistance may be enhanced by carefully selecting MMNC fabrication settings. Although more expensive than casting technologies like stir casting, the powder metallurgy approach shows promise for the production of high-quality MMNCs in large quantities. Stir casting is a low-cost and adaptable method. This method allows for complicated shapes to be cast using standard foundry methods. Nanoparticles have a high surface-to-volume ratio and poor wettability in metal melts, making them difficult to disseminate uniformly using traditional mechanical stirring methods.

Conclusions

- Microhardness values were found to be greater in Al/TiO_2 and Al/B_4C MMNCs than in the pure Al matrix. Microhardness measurements showed that Al/B_4C MMNCs were somewhat more durable than their Al/TiO_2 counterparts. With an increase in nanoparticle size and/or nanoparticle volume percentage, MMNC microhardness also rises.
- The corrosion resistance of Al/B_4C and Al/TiO_2 MMNCs was found to be much higher than that of the pure Al matrix when tested in a NaCl solution. The Al/TiO_2 MMNCs' CR was significantly lower than that of the Al/B_4C MMNCs.

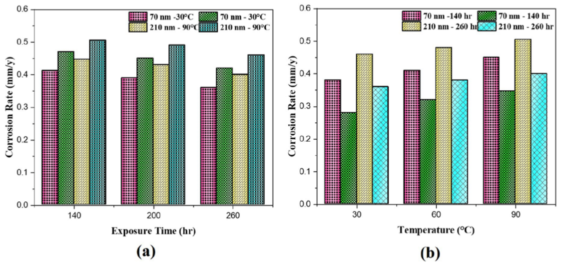

Figure 6: Calculated CR of Al/TiO_2 MMNCs varies depending of **(a)** Exposure time and **(b)** Solution temperature.

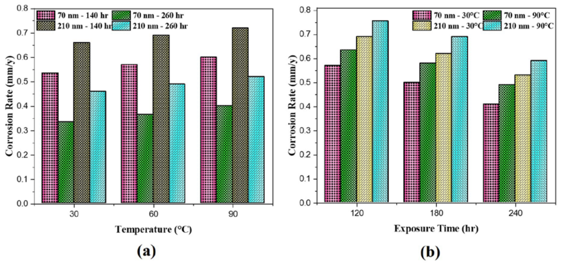

Figure 7: Calculated CR of Al/B_4C MMNCs varies depending of **(a)** Exposure time and **(b)** Solution temperature.

- When MMNCs are submerged in NaCl solution, their corrosion resistance declines as the size of the B_4C and TiO_2 nanoparticulates increases. In addition, the corrosiveness of MMNCs increases when the volume percentage of microparticles is raised over 4 vol.%. The nanoparticles' corrosion resistance decreases as the volume fraction increases because they are more prone to clump together.
- The CR of the MMNCs was shown to be statistically and physically related to the temperature of the NaCl aqueous solution and only weakly to the duration of its exposure to the solution.
- To assess the MMNCs' corrosion rates in varying NaCl solution conditions, mathematical correlations were created. Use of such correlations allows for rather accurate predictions of CR for Al/B_4C and Al/TiO_2 MMNCs.

Acknowledgements

None.

Conflict of Interest

None.

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