Research Article

The comparative study of conventional and ultra-high frequency induction sintering behavior of pure aluminum

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ARTICLE INFO	ABSTRACT
	In this study, compressibility, and conventional and ultra-high frequency induction sintering behaviors of 99.8% purity and 50-70 μm size range aluminum powders were investigated. In the compressibility studies, uniaxial-cold pressing method was used. Green samples were produced in the range of 50-275 MPa using different pressures. By measuring the apparent densities of the produced samples, the optimum compressibility pressure was determined as 200 MPa. Pure aluminum powder metal samples produced with this ideal pressing pressure were sintered in both classical and ultra-high frequency induction methods in the range of 500-600 ºC. Sintering was performed as 40 min in the traditional method and 5 min in the ultra-high frequency induction sintering method. As a result of the tests carried out in this study, it was determined that pure aluminum samples were successfully sintered with a high frequency induction system in a shorter time than traditional sintering method.

1. Introduction

Because of their superior strength to weight ratio, easy machining and excellent corrosion resistance, aluminum alloys and aluminum matrix composites (AMCs) have been applications in the automobile industry such as cylinder blocks, pistons and piston insert rings [1]. In addition to this, the aluminum alloys and AMCs are widely utilized in marine, aerospace and defence industries [2-5]. One of the manufacturing process of the AMCs is powder metallurgy (PM). When compared to other manufacturing processes likely conventional casting method, energy conservation can be increased up to 50% by using PM. Additionally, due to having final products via PM, there are nearly no raw materials [6]. Therefore, many researchers have focused their efforts on the study of the sintered AMCs.

Sintering of AMCs has some challenges. One of them is oxide layers. Aluminum powder have always oxide layers and these oxide layers prevent sintering. Eliminating these oxide layers is not possible with low temperature which is carried out for sintering AMCs. There are two processes most commonly used to decrease negative effect of the oxide layers on the sintering behavior and mechanical properties of the AMCs. First process is to increase contact areas among powders. For this purpose, higher compaction pressure is carried out to destroy oxide layers or some elements are added to create a liquid phase during sintering such as Cu [7-9]. In this way liquid phase can diffuse interfaces of powders. Hence interfacial bonding between powders is improved. Second process is to add elements which can help to decomposition aluminum oxide or reduce oxide [9-12]. Magnesium is very reactive and so it is used as a solid reducing agent. A possible reaction is

$$3Mg + 4Al_2O_3 \rightarrow 3MgAl_2O_4 \text{ (Spinel)} + 2Al \qquad (1)$$

Gokce and Fındık, in their study, investigated mechanical and physical properties of the Al-1%Mg sintered parts. This study shows that while the sintering of the wax added Al-1%Mg composite samples for dewaxing, large porosities were being formed and so volume increased, and density decreased. Consequently,

TRS of the wax added Al-1% Mg composite samples the increased as nearly 10 times in wax free samples [13].

Oh and Anh investigated effects of the Mg addition and compaction pressure on mechanical and microstructure properties of Al-3Cu composites. For this purpose, the compaction pressures of 100, 250, 500 MPa were carried out and Mg was added in three different compositions, 0.5, 1.5, and 2.5 wt.%. The study shows that because of the limited wettability of Mg element compared with Cu, the sintered density decreases with the increase of Mg addition. They reported that with the addition of small amount of Mg, the oxide layer is broken and also $MgAl_2O_4$ spinel structures are formed, so contact area between Al and Cu increases. As a result, small amount of Mg addition improves the mechanical behavior of Al-Cu composites [9].

Gokce et al. investigated effects of Mg content on mechanical properties and aging behavior of Al4CuXMg composites. For this purpose, four different premixed powder compositions, Al4Cu, Al4Cu0.5Mg, Al4Cu1Mg, Al4Cu2Mg, were used. Premixed powders were pressed at 400 MPa. Green compacts were sintered at 615 ºC for 1.5 h. After sintering, specimens were aged at 180 ºC. Aging time was varied between 6 h and 48 h. AS a result, highest hardness value was measured from 24 h aged Al4Cu2Mg alloy [14].

Boland et al. studied to improve the mechanical properties of P/M Al-Cu-Mg composites by using different Mg and Cu addition. In this study, 3.4, 4.4 and 5.4 wt.% Cu and 0.5, 1.5 and 2.5 wt.% Mg was added. Also, composites were compacted at pressures from 100 to 500 MPa and then sintered at various temperatures between 560 and 630 ºC and times between 1 and 100 min. It was reported that the optimum manufacture process for P/M 2324 (Al–4.4Cu–1.5Mg) included a compaction pressure 400 MPa followed by sintering at 600 ºC for 20 min [15].

Gokce et al. investigated mechanical properties of P/M pure Al, Al5CU and Al5Cu0.5Mg parts. Pure Al and premixed powders were pressed in uniaxially at 400 MPa. Pure Al compacts were sintered at 600 °C for 2 h. Al5Cu and Al5Cu0.5Mg composites were sintered at 590 °C for 90 min. The tests show that although the Al5Cu0.5Mg composites have the lowest density, they have the highest TRS and hardness [16].

Reducing the temperature and application time will also reduce the formation and thickness of the oxide layer [17]. Microwave, spark plasma and induction sintering are common sintering methods that reduce sintering temperature and time. Sintering of aluminum alloys and composites by microwave and spark plasma method has been studied in many aspects [18-22]. In their study, Zadra et al. Sintered pure aluminum with spark plasma sintering (SPS) method at 470-525 ºC temperatures. They reported that sintered samples had a hardness of 27 HV0.1 [23]. Zeng et al.

produced the pure Al P/M using SPS method. The sintering temperature range was chosen as 450-600 ºC. The highest hardness was determined as 37.7 HV in the samples sintered at 550 ºC [24]. Kwon et al. manufactured the P/M pure Al parts using SPS process at 280-560 ºC and reported that the relative density increased as the sintering temperature increased. [25]. Induction sintering method, another rapid sintering method, was widely studied on sintering of carbide and oxide ceramics such as WC, TiC, B_4C, Al_2O_3 and iron-based metallic parts. However, the sintering of aluminum-based parts has not become widespread [26-30]. Induction heating technology is used in production methods and stages such as heating, melting, welding and sintering [31]. In this technology, the heating parameters depend on the electromagnetic properties of the material to be heated. In induction sintering, ohmic heating takes place with the electromagnetic field changing with eddy currents. Skin Effect is the mechanism that concentrates the current density on the surface [32-34]. The relationship between electrical conductivity (σ), current frequency (f), magnetic permeability (μ) and skin depth (δ) is expressed in Eq. 2:

$$\delta = \frac{1}{\sqrt{\sigma \pi f \mu}} \qquad (2)$$

Induction sintering is a widely used method for the rapid sintering of iron-based P/M parts due to its electromagnetic properties. With this study, it was aimed to reveal the sinterability of aluminum based powder metal parts by induction fast sintering. For this purpose, the P/M pure aluminum green samples were manufactured by cold pressing method, by applying 200 MPa and sintered by using 40 minutes conventional and 5 minutes ultra-high frequency induction method with 11 different temperatures between 500-600 ºC temperatures. When comparing the test and analysis results of the samples sintered with the conventional and ultra-high frequency induction sintering (UHFIS) method, it was found that aluminum alloys were successfully sintered in a much shorter time with the ultra-high frequency induction sintering method.

2. Materials and Method

In this study, pure aluminum powders with 99.8% purity and 50-70 μm powder size range were used. The compressibility of aluminum powders was first investigated. For this purpose, aluminum powders prepared by weighing on a scale with a sensitivity of 0.0001 gr were uniaxial cold pressed via hydraulic press. In the compressibility analysis, pressing was performed in the pressure range of 50-275 MPa with 25 MPa intervals. After pressing, the density of the samples was calculated in accordance with the Archimedes principle [32].

The 11 green samples pressed at 200 MPa compaction pressure determined as a result of compressibility tests have

traditionally been sintered in the ash furnace. In the other 11 green samples, ultra-high frequency induction sintering was applied using 8 kW power and 900 kHz high frequency induction heating machine. n the induction and conventional sintering methods, 500-600 ˚C was chosen as the sintering temperature range with 10˚C intervals. In sintering of the green samples, the application time was 40 minutes in the traditional method. In ultra-high frequency sintering, the sintering time was 5 minutes. The image of the samples after sintering can be seen in Figure 1.

Sintered densities of the samples were measured by the Archimedes principle. The sintered density is defined in Eq. 3:

$$\rho = \frac{m_a}{m_c - m_w} x \rho_{water} \qquad (3)$$

Where:

m_a is weight of sintered part in air, m_c is weight of sintered part in air after submerged from water, m_w is weight of sintered part in water.

The samples were sanded with 180-1200 grid sandpaper respectively, polished with 3 and 1 micron diamond solutions via Metkon Farcipol 1V polishing machine. The polished samples were etched with Keller solution (2.5 ml HNO_3, 1.5 ml HCl, 1 ml HF, 95 ml water). The metallographic examinations were carried out via Nicon Eclipse LV150N optic microscope. The Bulut Makine BMS 200 RB hardness tester was used for the Brinell hardness measurements of the samples. Hardness measurements were carried out in accordance with ASTM E10-15a standards [35]. In the Brinell hardness tests, 62.5 kg load was applied, and 2.5 mm diameter steel ball indenter was used. Measurements were repeated 5 times for each sample and these 5 measurements were averaged.

3. Results and Discussions

To determine the compressibility of 2.5 and 5 g pure aluminum powders, the powders were pressed using the uniaxial cold pressing method in the pressure range of 50-275 MPa. Compressibility graphs of pressed samples obtained by density measurements performed with the Archimedes principle are shown in Figure 2. As is well known, aluminum powders have higher compressibility than iron alloy powders [36]. Therefore, it is possible to achieve high green density in P/M aluminum parts with lower compacting pressures. [37]. In the 50-175 MPa compaction pressure range, the density was found to be about 2.40 to 2.5 g/cm^3. The substantial increase in density occurred when the compaction pressure increased from 175 MPa to 200 MPa. Even after the compaction pressure was increased up to 275 MPa after 200 MPa, there was no significant increase in density values. This result showed that the ideal compressibility for the aluminum powders used was at a compaction pressure of 200 MPa.

Figure 3 shows the weight changes depending on the sintering temperature in the samples pressed with 200 MPa compaction pressure and then sintered with traditional and UHFIS methods. There were two main reasons for weight change in aluminum powder metal samples. The first was the weight increase observed as a result of oxidation. Another was the weight reductions that occurred as a result of evaporation of paraffin-based mold lubricants or lubricants added to the powder mixture that penetrated the powder metal sample surface during evaporation. In this study, no lubricant or binder was added to the powder mixture. However, solid paraffin lubricants have been used to prevent aluminum powders from plastering on the mold walls, which is a common problem in cold pressing of aluminum powders. These lubricants were determined to penetrate the un-sintered sample surfaces after pressing. Compared to the UHFIS method of the traditional method, the lubricant gave more successful results in flying. The main reason for this was that the traditional sintering time was much longer than UHFIS. Another reason for this was that the electromagnetic properties required for induction heating were not found in the solid paraffin lubricant.
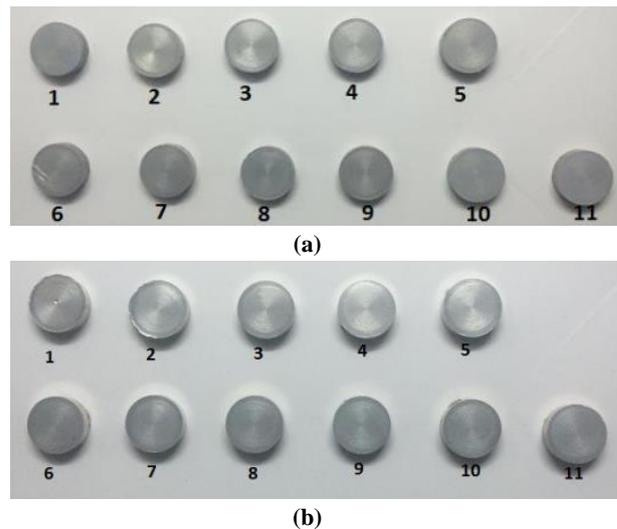

(a)

(b)

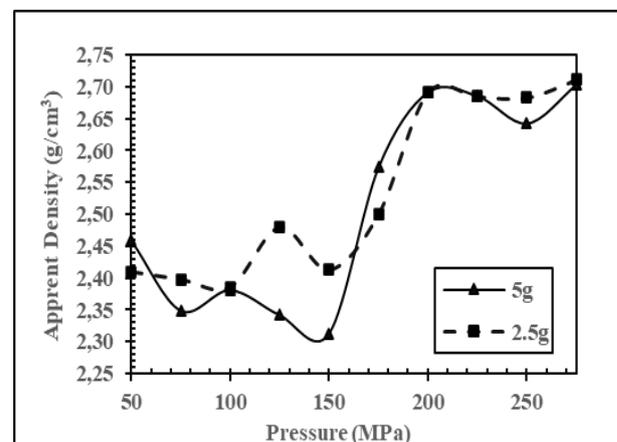Figure 1. Sintered Samples with Traditional (a) and UHFIS (b) Methods

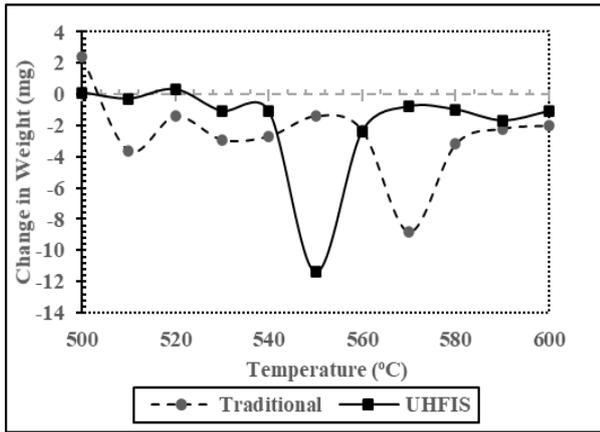Figure 2. Graph of density as a function of compacting pressure

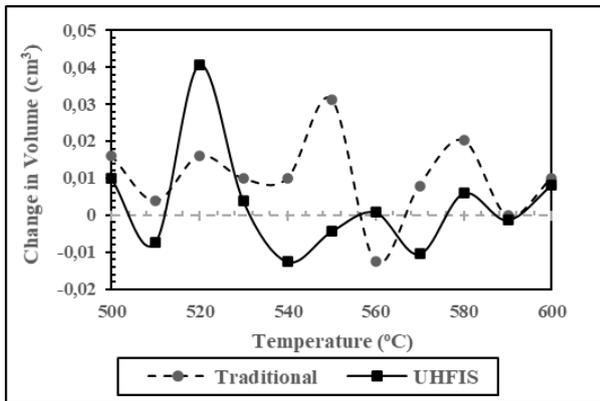Figure 3. Change in weight as a function of sintering temperature

Figure 4. Change in volume as a function of sintering temperature

The volume changes depending on the sintering temperature in the samples sintered with traditional and UHFIS methods are shown in Figure 4. In powder metal samples, volume increase may occur due to grain growth, flying lubricant and binder, swelling caused by phase transformations. The main mechanism for the decrease in volume is that the pores are closed by sintering after pressing. The lowest volume change was observed in UHFIS at 540 ℃, while the traditional method generally showed an increase in volume. However, a decrease in volume was determined in the sintering process performed at 560 ℃.

The density changes depending on the sintering temperature in the samples sintered with traditional and UHFIS methods are given in Figure 5. The samples with the highest density obtained in samples sintered by UHFIS method were samples sintered at 540 ℃. The lowest density values were measured in the samples sintered at 560 ℃. In the sintering carried out in the traditional method in the 520-580 ℃ temperature range, the density values were measured close to each other, in the range of approximately 2.65-2.66 gr/cm³. In the traditional method, the lowest density was determined in the sample sintered at 500 ℃. In general, in the UHFIS method, the apparent density was obtained higher than the traditional method. This result shows that the UHFIS method, where heating with the electromagnetic mechanism takes place, is effective in closing the pores even in a shorter time compared to conventional sintering. Thus, it can be said

that the UHFIS method has a positive effect on the sinterability of aluminum.

The hardness changes of the samples sintered by traditional and UHFIS methods depending on the sintering temperature are given in Figure 6. Higher hardness values were obtained in samples sintered by the UHFIS method compared to the traditional method. When the density changes depending on the sintering temperature (Fig. 5) and the hardness changes (Fig. 6) are examined together, it is understood that for the traditional method, the samples are insufficiently sintered at 500 ℃ sintering temperature. In these samples sintered by the traditional method, while the hardness increased up to 550 ℃, as a result of the grain size, it decreased again by about 14% after 550 ℃. Although the highest hardness value was measured at 500 ℃ in samples sintered by UHFIS method, when the density and hardness values of 540 ℃ sintered samples are examined together, it can be said that this temperature is the ideal sintering temperature of aluminum samples for the UHFIS method.

Figure 7 shows the microstructures of sintered samples at 520, 540, 560, 580 and 600 ℃ by UHFIS method, respectively. Microstructure images were obtained with an optical microscope at 500x magnification.
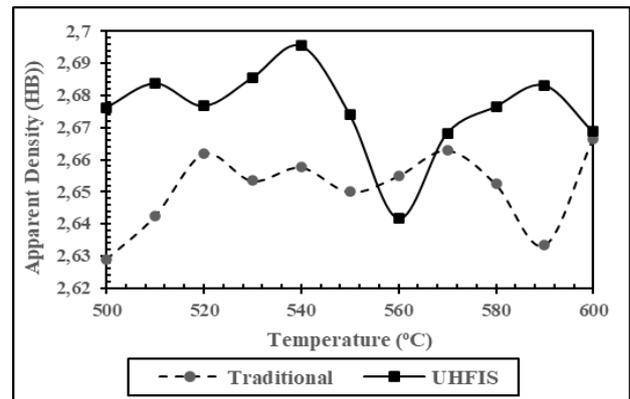

Figure 5. Apparent density of sintered samples as a function of sintering temperature
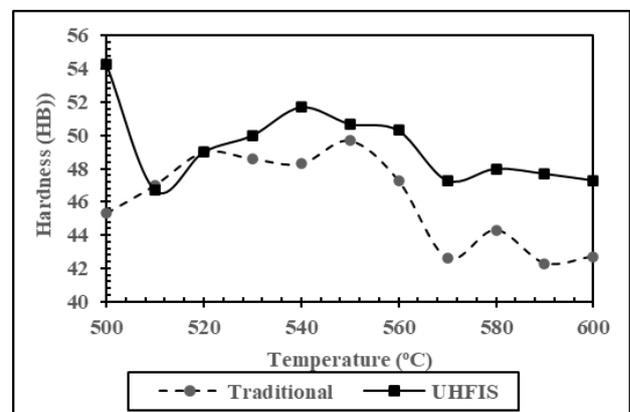

Figure 6. Hardness of the sintered samples as a function of sintering temperature
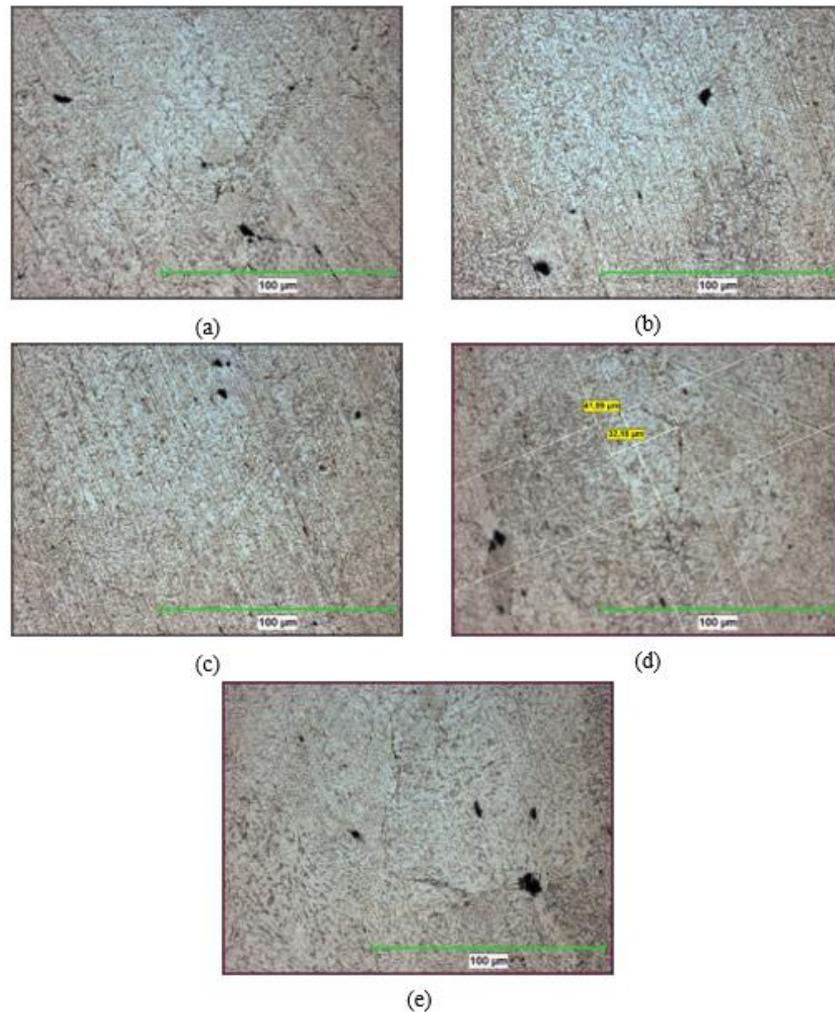
Figure 7. Microstructures of sintered samples at various temperatures by UHFIS Method: a) 520 °C, b) 540 °C, c) 560 °C, d) 580 °C and e) 600 °C.

When the microstructure images were examined, it was seen that after the sintering, the number of pores between the particles was considerably decreased and the existing pores were small in size. Besides, the effect of eddy currents in the microstructure of the sample sintered at 600 °C was determined.

In Figure 8, microstructure images of the samples sintered by traditional and UHFIS methods are given at 600 °C, respectively. Images were taken under an optical microscope at 200x magnification. These microstructure images show differences in the size and distribution of the pores of these two samples with similar density. In the traditional method, the pores after sintering were much larger than the pores in the samples sintered by the UHFIS method. In addition, it was determined that these pores showed a more homogeneous distribution in the samples sintered by the UHFIS method. This indicates the sintering behavior and success of P / M pure aluminum with the UHFIS method.
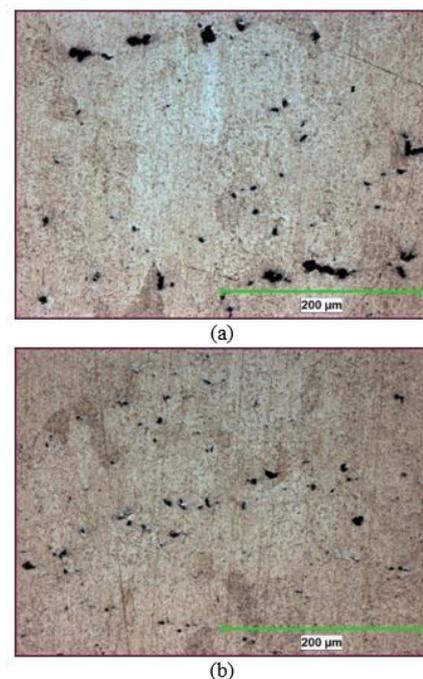

Figure 8. Microstructures of sintered samples with (a) traditional and (b) UHFIS Methods at 600 °C

4. Conclusions

In this study, compressibility of uniaxial cold pressing with pure aluminum powders having 99.8% purity and 50-70 μm size was investigated. Traditional and ultra-high frequency induction sintering behaviors of samples pressed with optimum compaction pressure determined by compressibility tests were compared. Sintering processes were carried out with both methods at 10 ℃ intervals in the range of 500-600 ℃. In the traditional method, sintering was performed as 40 minutes. In the UHFIS method, sintering was applied for 5 minutes. The results are as follows:

• The optimum compaction pressure of pure aluminum powders with 99.8% purity and 50-70 μm size is 200 MPa.

• Traditional method is more successful in evaporating lubricant than UHFIS method.

• Density values of samples sintered by UHFIS method are higher than density values of samples sintered by traditional method.

• The hardness values of the samples sintered by UHFIS method are higher than the hardness values of the samples sintered by the traditional method.

• The UHFIS method decreases the pore size more effectively and the distribution of the pores in the internal structure becomes more homogeneous.

• Pure aluminum powders, 5 min the optimum sintering temperature is 540 ℃ during sintering with UHFIS method.

• Thanks to the work done, it was shown that it was successfully sintered to pure aluminum powder metal samples in less than 8 times less time by using induction.

Declaration

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The author(s) also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

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