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Wear behavior of NiTi+La_x (x:1, 3, 5 wt.%) alloys produced via M/A, P/M and Boro-Sintering techniques

M/A, T/M ve Boro-Sinterleme teknikleriyle üretilen NiTi+La_x (x:1, 3, 5 % ağı.) alaşımlarının aşınma davranışları

Author(s) (Yazar(lar)): Ceylan KARABUDAK¹, Nimet KARDEŞ SEVER², Hasan DURAN³, Sinan AKSÖZ⁴

ORCID¹: 0000-0002-3805-0703

ORCID²: 0000-0002-3395-0171

ORCID³: 0000-0002-0605-8882

ORCID⁴: 0000-0003-4324-5043

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Wear Behavior of NiTi+La_x (x:1, 3, 5 wt. %) Alloys Produced via M/A, P/M and Boro-Sintering Techniques

Highlights

- ❖ NiTi+La_x alloys were produced by M/A, P/M, and Boro-Sintering techniques.
- ❖ Wear behavior of NiTi+La_x alloys (x: 1, 3, 5 wt. %) was examined.
- ❖ FESEM and EDS analyses were conducted.

Graphical Abstract

This study investigated the wear behavior of the NiTi+La_x (x: 1, 3, 5 wt. %) alloys produced via P/M.

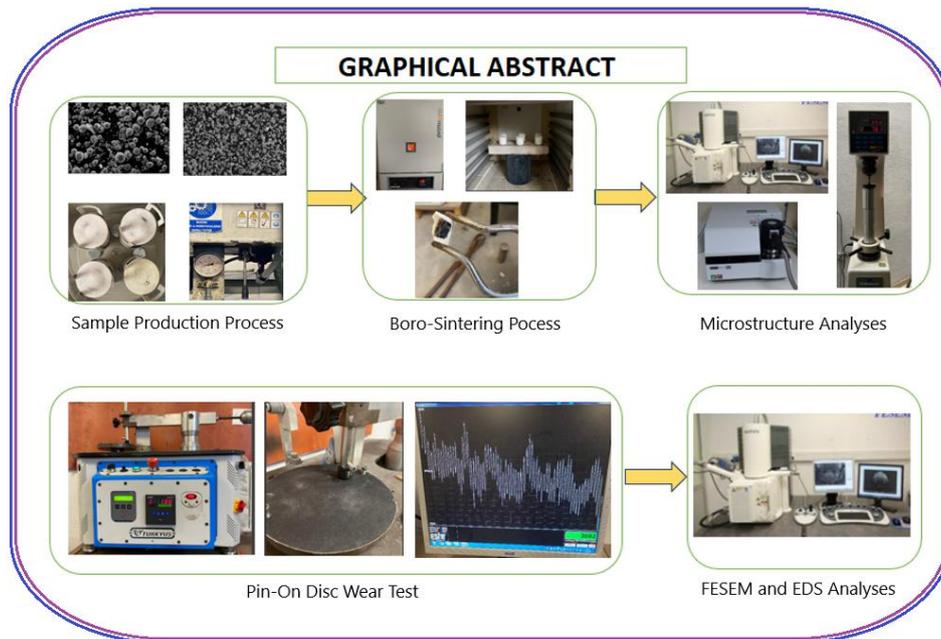


Figure. Graphical abstract

Aim

The main objective was to determine whether increasing La content increases the wear resistance in NiTi+La_x alloys.

Design & Methodology

FESEM, EDS, and wear tests were conducted on NiTi+La_x alloys produced by M/A, P/M, and the Boro-Sintering method.

Originality

This study was carried out because more research and publications on NiTi+La_x alloys are needed.

Findings

This production method directly affected the wear properties. The best wear properties were obtained in the NiTi+La_x alloy with 1wt.%La addition.

Conclusion

It was observed that La element could be added to NiTi alloy by P/M techniques and it could increase the wear resistance.

Declaration of Ethical Standards

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Wear Behavior of NiTi+La_x (x:1, 3, 5 wt. %) Alloys Produced via M/A, P/M and Boro-Sintering Techniques

Araştırma Makalesi / Research Article

Ceylan KARABUDAK¹, Nimet KARDEŞ SEVER², Hasan DURAN³, Sinan AKSÖZ³

¹Faculty of Technology, Metallurgical and Materials Engineering Department, Pamukkale University, Türkiye

²Faculty of Technology, Automotive Engineering Department, Pamukkale University, Türkiye

³Faculty of Technology, Metallurgical and Materials Engineering Department, Pamukkale University, Türkiye

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ABSTRACT

The powder Metallurgy (P/M) method is mainly preferred in the production of superior metal alloys that cannot be produced with traditional methods. In this study, the first step was mechanical alloying (M/A) of NiTi+La_x powders via ball milling. Then the alloyed powders were pressed and gradually sintered using airtight ceramic crucibles filled with boron salt (for alloying and creating an atmospheric environment) to obtain NiTi+La_x alloys. The wear properties and behaviors of the samples were determined (with different La amounts (1wt.%, 3wt.%, and 5wt.%) by pin-on-disc testing. The experiments were carried out at a sliding speed of 1.2 m/s and under 5N, 10N, and 15N loads. The microstructures of the worn surfaces were characterized by FESEM and the distribution of elements was analyzed by EDS. As a result, the lowest friction coefficient and wear loss were observed in the NiTi+La_x alloy containing 1wt.% La. The amount of wear volume increased with the increase in load and La content.

Keywords: NiTi+La_x Alloys, Boro-Sintering of NiTi-La Alloys, Wear Behaviour of NiTi+La_x Alloys, Powder Metallurgy (P/M), Mechanical Alloying (M/A).

M/A, T/M ve Boro-Sinterleme Teknikleriyle Üretilen NiTi+La_x (x:1, 3, 5 % ağı.) Alaşımlarının Aşınma Davranışları

ÖZ

Toz Metalurjisi (T/M) yöntemi, geleneksel yöntemlerle üretilmeyen üstün metal alaşımlarının üretiminde çoğunlukla tercih edilmektedir. Bu çalışmada, ilk adım NiTi+La_x tozlarının bilyalı öğütme yoluyla mekanik alaşımlanması (M/A) olmuştur. Daha sonra alaşımlı tozlar preslenmiş ve bor tuzu ile doldurulmuş hava geçirmez seramik potalarda (alaşımlama ve atmosferik ortam oluşturmak için) kademeli olarak sinterlenerek NiTi+La_x alaşımları elde edilmiştir. Numunelerin aşınma özellikleri ve davranışları (farklı La miktarlarıyla (x: 1,3,5 % ağı.)) disk üstü pim testi ile belirlenmiştir. Deneyler 1,2 m/s kayma hızında ve 5N, 10N ve 15N yükler altında gerçekleştirilmiştir. Aşınmış yüzeylerin mikro yapıları FESEM ile karakterize edilmiş ve elementlerin dağılımı EDS ile analiz edilmiştir. Sonuç olarak, en düşük sürtünme katsayısı ve aşınma kaybı ağırlıkça %1 La içeren NiTi+La_x alaşımında gözlemlenmiştir. Aşınma hacmi miktarı yük ve La içeriğindeki artış ile artmıştır.

Anahtar kelimeler: NiTi+La_x Alaşımları, NiTi-La Alaşımlarının Boro-Sinterlenmesi, NiTi+La_x Alaşımlarının Aşınma Davranışı, Toz Metalurjisi (T/M), Mekanik Alaşımlama (M/A).

1. INTRODUCTION

Materials science is constantly growing with the development of new and innovative materials. P/M applications are preferred over the other methods due to their ability to produce complex-shaped and high-performance parts with superior properties [1-3]. NiTi Shape Memory Alloys (SMAs) are one of the most important material technologies that are generally used in automotive, biomedical, optical, and micro-electromechanical systems, and space applications due to their shape memory and superelasticity properties. Since

material technology is constantly evolving and changing, new areas of application are created by adding different elements to these existing materials [4].

Rare-earth elements (REE) are a group of seventeen elements located in the periodic table. REE have become extremely important to material technology thanks to their unique magnetic, phosphorescent, and catalytic properties since the beginning of the 1990s. Lanthanum (La), one of the REE, is utilized in catalysts, magnetic materials, glass and ceramic materials, precision devices, optical applications, and biomedical applications. Its use is increasing especially in energy technologies, green energy, and advanced technology products. [5-7].

*Sorumlu Yazar (Corresponding Author)
e-posta : ceylankarabudakck@gmail.com

P/M is one of the most appropriate methods to produce metal alloys to improve the properties of an existing pure metal. Although there are many methods, M/A method is a frequently preferred method in P/M applications since it provides a more homogeneous structure by subjecting different metal powders to a high-energy ball milling process in a fracture-welding cycle [8].

NiTi alloys are used in actuators in the aviation field due to their high-stress ratio, better flexibility, and fatigue resistance. La addition to NiTi SMA can improve the mechanical properties and optimize the material for a wide range of applications. For this reason, the addition of La to NiTi alloys is becoming an important research topic. For example; it can increase mechanical strength, improve wear resistance, and extend the service life [9]. According to DIN 50320, wear is defined as an undesirable change that occurs due to the separation of small particles from the material surface as a result of mechanical effects. 50% of the damage to machines is caused by wear. Thus, wear resistance is an important material property [10-12]. When literature studies about NiTi alloys produced with the P/M method are examined, it is seen that the effect of temperature on wear behavior has been investigated. However, the effects of alloying elements such as La alloy added to NiTi alloys, which directly affect the transformation and mechanical properties, have not been investigated. Thanks to this study, the importance of producing new NiTi alloys with new techniques, known as innovative areas in the literature, has been emphasized. A pioneering study has been created in this field thanks to the study [13-20]. In this study, the wear behavior of the NiTi+La_x alloys, produced via P/M, was investigated.

2. MATERIAL AND METHOD

In this study, high purity pre-alloyed NiTi powders were subjected to the mechanical alloying process using different amounts of La powders (1wt.%, 3wt.%, and 5wt.%). MA process was conducted using an SFM-1 Desk-Top Planetary machine which is located in the Mechatronics Engineering Department of Pamukkale University. The average grain size of the NiTi powders is approximately 40 µm. La, one of the REE, has a grain size of approximately 70 µm. The alloying was carried out in two directions for 300 min. After the microstructure and element analysis, samples were pressed and boro-sintered to create a permanent and homogeneous alloying between NiTi and La powders. The details of the production process are described in the study conducted by Karabudak et al. [18]. It was reported in the literature that the B/S process increases the wear resistance of the sample.

Pin-on-disc type dry wear test was performed to investigate the wear behavior of these new NiTi+La_x alloys [19]. Wear tests were performed under dry conditions (no lubricant) with Turkeyus brand pin-on-disc machine (see Figure 1) in accordance with ASTM G99-05 standards. Cylindrical wear samples with a diameter of 10 mm were tested under 5N, 10N, and 15N normal

loads at a sliding speed of 1.2 m/s for 120 seconds. The disc was cleaned with pure alcohol before the wear test. Sandpaper (grit size 600) was used as the abrasive material. The samples were placed perpendicularly to the sandpaper disc. The density of NiTi alloy is 6.45 g/cm³ and the density of La is 6.15 g/cm³. The density of NiTi+La_x alloy was assumed to be near 6.17 g/cm³ for all samples.

The wear behavior together with the load applied to the sample was recorded to calculate the friction coefficient. The equation below was used to calculate the friction coefficient.

$$\text{Coefficient of Friction } \mu = F/P \quad (1)$$

In the equation, F and P indicate the friction and the normal forces respectively.

The volume loss value from the weight loss data is found with the following equation.

$$\text{Volume Loss (mm}^3\text{)} = \text{Weight Loss (g)} / \text{Density (g/mm}^3\text{)} \quad (2)$$

The specific wear rate formula is as follows.

$$\text{Specific Wear Rate (mm}^3\text{/Nm)} = \text{Volume Loss (mm}^3\text{)} / (\text{Sliding distance (m)} \times \text{Load (N)}) \quad (3) \quad [20]$$

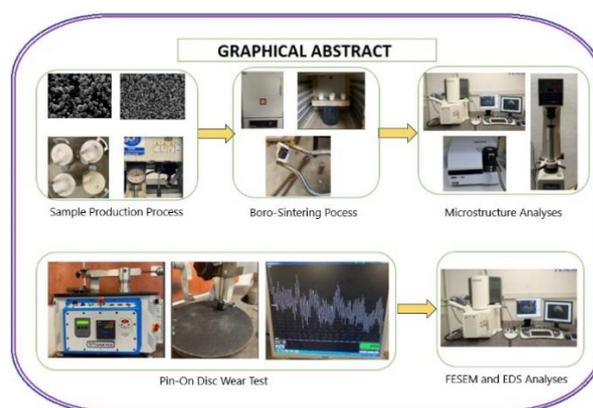


Figure 1. Graphical abstract

After the completion of wear tests, the worn surfaces were examined through FESEM and EDS.

3. RESULTS AND DISCUSSION

In Figure 2, the FESEM images of NiTi+La_x (1wt.%, 3wt.%, and 5wt.%) materials after 144 m sliding distance at 15N load are shown respectively. The EDS analyses of worn sample surfaces are shown. It increases with the La ratio, and an increased abrasion ratio, deep grooves, and defective wear surface were seen in the FESEM images. In the EDS results, the B and Na content originated from Borax. The lowest intensity of the C content came from the sandpaper disc [21]. O impurities were seen in all of the samples since the worn surfaces were directly oxidized. This oxide structure is directly related to the sintering temperature, time, and the sintering environment [22, 23]. As the La content increased, the amount of oxides also increased. The oxides could create an effect similar to lubrication on the surface; therefore, they could alter the wear properties [24, 25]. The oxygen content on the surface increased with the increasing La content. In the alloyed NiTi alloys, heat treatments at

1000°C increased the TiO₂ oxidation layers, which also directly affected the wear properties [26, 27]. According to the EDS analysis, oxygen (O) was identified as the most abundant element with a concentration of 33.28 wt.%, followed by titanium (Ti) at 23.50 wt.%. The presence of these elements is attributed to surface oxidation resulting from sintering processes and wear mechanisms (In Figure 2.c). The La elements could be clearly detected in all of the samples due to the effects of M/A, B/S, and sintering techniques. The porosity occurred due to the production methods and directly affected the mechanical properties [28].

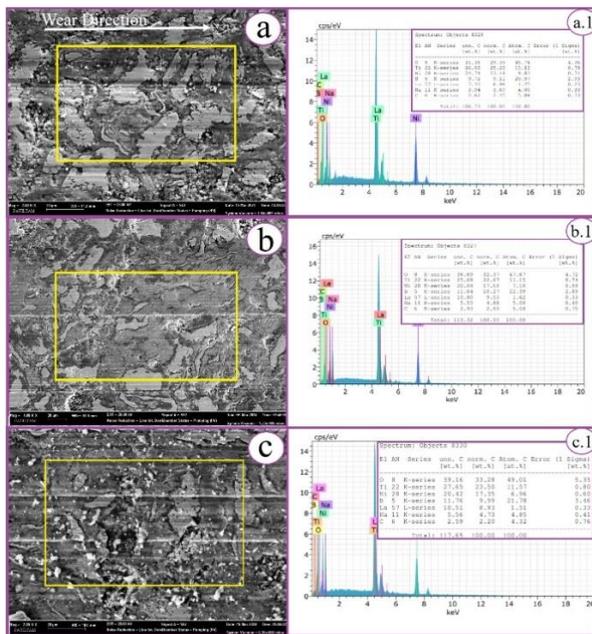


Figure 2. Worn surfaces FESEM and EDS analyses results as; a) NiTi+ 1 wt.% La alloy, a.1) EDS analysis, b) NiTi+ 3 wt.% La alloy b.1) EDS analysis, c) NiTi+ 5 wt.% La alloy, c.1) EDS analysis

After the sample production was carried out, the hardness of the samples were measured in HRC with the Schimutzu device at Pamukkale University. 5 measurements were taken for each sample. The final results were based on the average of these 5 measurements. An increase in hardness was observed in the materials with an increasing %La ratio. The highest value was 47.8 HRC in the NiTi+ 5 wt.% La alloy (Table 1). In conclusion, oxide formation plays a direct role in hardness, and it was observed that hardness increases with oxide formation [29, 30].

Table 1. HRC hardness data of NiTi+La_x alloys

%La Rate	Average (HRC)
%1 La	28.6 HRC
%3 La	41.6 HRC
%5 La	47.8 HRC

Table 2 shows the weight loss data of NiTi+ 1 wt.% La, NiTi+ 3 wt.% La, and NiTi+La5wt.% samples measured under different loads. Weight loss increased with increasing load. The highest weight loss was seen in the

NiTi+ 5 wt.% La sample under a 15N load. It can be said that the increase in La content negatively affected the wear properties. The best wear properties were observed in NiTi+ 1 wt.% La alloy. These results suggested that the amount of La contributes to wear resistance up to a certain level (probably between 1% and 3%), but when it reaches 5%, the material becomes brittle and the wear resistance decreases.

Table 2. Post-wear weight loss data of NiTi+La_x alloys under 5N, 10N and 15N

Test Nr.	Sample	Force (N)	Before the wear test (g)	After the wear test (g)	Weight loss (g)
1	NiTi+ 1 wt.% La	5	2.8938	2.8876	0.0062
2	NiTi+ 1 wt.% La	10	2.8876	2.8745	0.0131
3	NiTi+ 1 wt.% La	15	2.8745	2.8574	0.0171
4	NiTi+ 3 wt.% La	5	2.8113	2.8049	0.0064
5	NiTi+ 3 wt.% La	10	2.8049	2.7918	0.0131
6	NiTi+ 3 wt.% La	15	2.7918	2.7704	0.0214
7	NiTi+ 5 wt.% La	5	2.9224	2.9158	0.0066
8	NiTi+ 5 wt.% La	10	2.9158	2.8970	0.0188
9	NiTi+ 5 wt.% La	15	2.8970	2.8625	0.0345

In Figure 3, volume loss increased in the samples as the load increased. The highest volume loss was observed in the NiTi+ 5 wt.% La alloy sample. With the increase in La content, the wear properties worsened. It was seen that the increase in the oxide ratio of the internal structure was directly proportional to the La content. Thus, oxidation caused a direct increase in volume and weight losses. [31]. It could be said that the B/S process increased the wear resistance of the sample. When Figure 3 was examined for the NiTi+ 1 wt.% La alloy, it could be observed that the volume loss increased in direct proportion to the load. While the highest volume loss was 2.771 mm³ at 15N load, the lowest volume loss was 1.004 mm³ at 5N load. In the NiTi+ 3 wt.% La alloy, the highest volume loss was 3.468 mm³ at 15N load, while the lowest volume loss was 1.037 mm³ at 5N load. In the NiTi+ 5 wt.% La alloy, the highest volume loss was 5.591 mm³ at 15N load, and the lowest volume loss was 1.069 mm³ at 5N load.

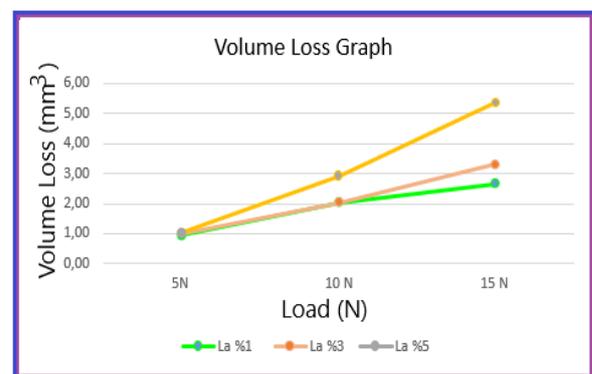


Figure 3. Post-wear volume loss graph of NiTi+ 1 wt.% La, NiTi+ 3 wt.% La, NiTi+ 5 wt.% La samples. The specific wear rate of NiTi+ 1 wt.% La, NiTi+ 3 wt.% La, NiTi+ 5 wt.% La samples was shown in Figure 4. For the NiTi+ 1 wt.% La sample, the wear rate initially

increased with load and then decreased, since the change in load was larger than the change in volume loss. For the NiTi+ 3 wt.% La and NiTi+ 5 wt.% La samples, an increase in wear rate was observed with increasing load.

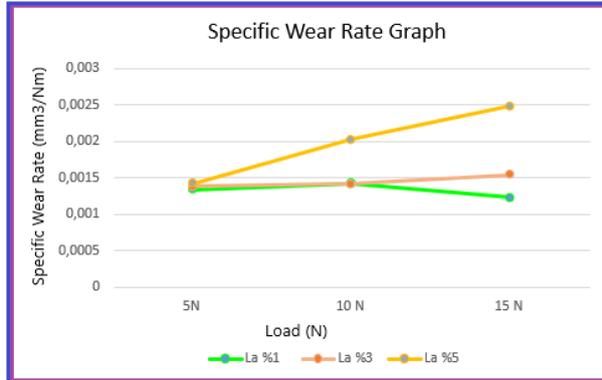


Figure 4. Specific wear rate graph of NiTi+ 1 wt.% La, NiTi+ 3 wt.% La, NiTi+ 5 wt.% La samples after wear

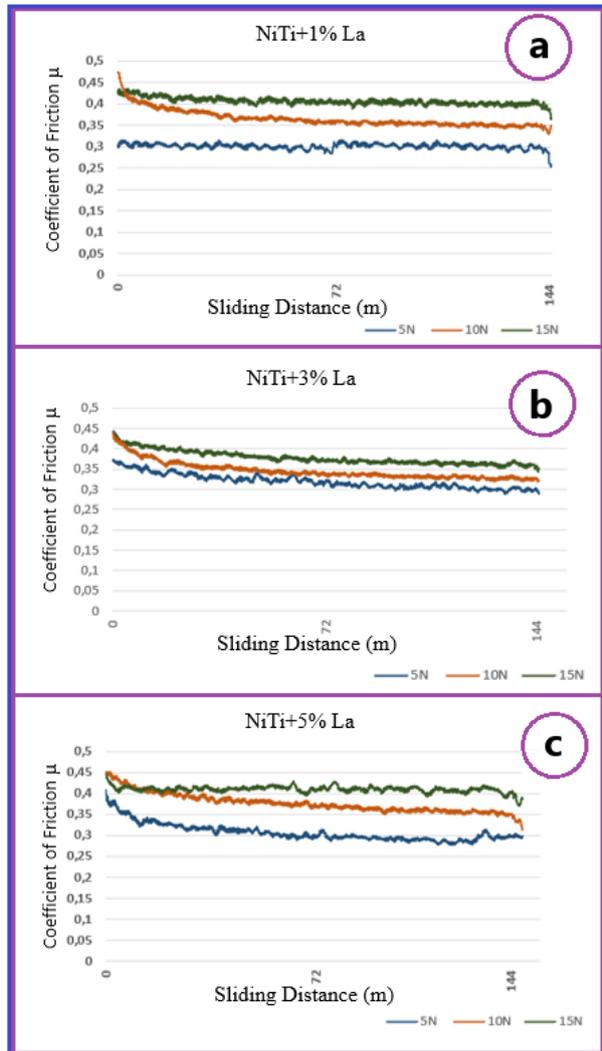


Figure 5. Friction coefficient graph of a) NiTi+ 1wt.% La, b) NiTi+3 wt.% La, c) NiTi+ 5 wt.% La samples

Figure 5 shows the coefficient of friction values for NiTi+ 1 wt.% La, NiTi+ 3 wt.% La, NiTi+ 5 wt.% La samples. In the NiTi+ 1 wt.% La sample (Figure 5.a), the friction coefficient value increased with the increase in load, and the highest friction coefficient value was observed as 0.425 in the test conducted under 15N load. For the NiTi+ 3 wt.% La (Figure 5.b) and the NiTi+ 5 wt.% La (Figure 5.c) samples, the friction coefficient increased with the increasing load, and the highest friction coefficient was approximately 0.450 under 15N load [29]. These results were found to be similar to the literature [32].

4. CONCLUSION

The effect of La addition on the wear behavior of NiTi+La_x alloys was investigated in this study. The NiTi and La powders were alloyed using P/M, M/A, and B/S methods. The following findings were obtained during the study;

- FESEM images showed that the highest wear effects were seen in the sample with 5% La content, and the best (lowest) wear surface was seen in the alloy with 1% La content. The porosity occurred with the increase in La ratio in the NiTi alloys and directly affected the wear properties.
- When EDS analyses were examined, it was seen that the O content increased depending on the increased La ratio in all samples. This was interpreted as the oxide formation activity of the La content. This oxide layer affected wear properties directly. The highest content of O was 33.28 wt.% for the NiTi+ 5 wt.% La.
- When NiTi+ 1 wt.% La, NiTi+ 3 wt.% La, NiTi+ 5 wt.% La alloys were tested in wear tests under 5N, 10N, and 15N loads with the sliding distance kept constant at 144m. An increase in volume loss was observed with increasing % La ratio and load. While the highest volume loss was 5.591 mm³ in the NiTi+ 5 wt.% La sample under 15N load, the lowest volume loss was calculated as 1.004 mm³ in the NiTi+ 1 wt.% La sample under 5N load.
- In EDS results; the La elements were clearly detected, because of the effects of M/A, B/S, and sintering technique.
- The specific wear rate for the NiTi+ 1 wt.% La alloy initially increased under 5N and 10N and then decreased under 15N load. The wear rate also increased with increasing load for the NiTi+ 3 wt.% La, NiTi+ 5 wt.% La alloys. It could be due to the increase in the hardness of the material with the % La ratio.
- The friction coefficient increased with increasing load. The lowest friction coefficient was approximately 0.3 in NiTi+ 1 wt.% La alloy for 5N load. The highest friction coefficient was

approximately 0.45 under 15N load in NiTi+ 3 wt.% La and NiTi+ 5 wt.% La samples.

In conclusion, it was possible to successfully produce high-quality and well-defined NiTi+La_x alloys utilizing M/A, P/M, and B/S techniques in the study. All of the processes contributed to the production of novel and high-performance materials. Additionally, this production method directly affected the wear properties. The best wear properties were achieved in the NiTi+ 1 wt.% La alloy. The increase in the La ratio negatively affected the properties.

ACKNOWLEDGEMENT

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DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Sinan AKSÖZ: He designed the experiments, analyzed the results, and performed the writing process.

Nimet KARDEŞ SEVER: She analyzed the experimental results, and performed the writing process.

Hasan DURAN: He conducted the experiments and analyzed the results.

Ceylan KARABUDAK: She conducted the experiments, and performed the writing process.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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