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The Effect of Heat Sterilization on the Surface Topography and the Tensile Properties in Various Nickel Titanium Wires Including a Korean Product

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The purpose of this study is to investigate the changes of mechanical properties and surface topography of various nickel titanium wires after heat sterilization for recycling with quantitative method. The materials used were four kinds of nickel titanium orthodontic wires including a Korean product.

Experimental specimens were treated with two kinds of heat sterilization methods ; dry heat (180℃, 60min) and autoclave (121℃, 15-20psi, 30min). Mechanical properties were evaluated by tensile test with Instron 4466 (load cell capacity :1000 kg, cross head speed : 5mm/min, grip distance:40mm, in room temperature). Surface topography of various wires was compared with each other qualitatively by using scanning electron microscopy and quantitatively by using profilometer. The findings were analyzed statistically with student t-tests.

The results were as follows;

1. Neither method of heat sterilization had any effects on tensile properties of the nickel-titanium wires used in this experiment.
2. Before heat sterilization, the surface smoothness was highest in Optimalloy, followed by Align and Sentalloy, with NiTi showing the lowest smoothness value.
3. In surface topography, Align and Optimalloy were not influenced by heat sterilization. NiTi, on the other hand, had increased roughness after dry heat sterilization and Sentalloy showed the same tendency after each of the two heat sterilization procedures.

Key Words : dry heat sterilization, autoclave, surface topography, nickel titanium wire

While nickel-titanium wires exhibit excellent elasticity and low stiffness, their high cost have compelled some clinicians to reuse them. Buckthal et al.[1] mentioned that about 52% of the clinicians who use nickel-titanium wires recycle them. It would be useful, then, to find out whether approved sterilization procedures cause changes in mechanical properties of the wires.

There had been some studies which demonstrated the physical changes following clinical use of orthodontic arch wires[2, 3]. Sarkar demonstrated in his study that nickel titanium wire had a tendency to corrode when subjected to either oral fluids or chloride solutions[7]. Schwaninger, on the other hand, found no changes in the load deflection characteristics of Nitinol following long-term immersion in a 1% NaCl solution[8]. Mayhew

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Table 1. Materials used in investigation

Material	Manufacturer	Address
Align	A-Company	San Diego CA 92131-1683 U.S.A.
Niti	Ormco	1332 S Lone Hill Av, Glendora, CA 91740, U.S.A.
Optimalloy*	Jinsung Ind. Co	137-1 Ojeon-dong Euiwang-si Gyeongki-do 437-070, Korea
Sentalloy	Tomy	Shinwa BLDG., 15-1, Uchikanda, 3-chome, Chiyoda-ku, Tokyo 101, Japan

* The commercial name has been changed from Ortholloy® of LG-cable since 1998.

observed no detrimental changes in either the mechanical properties or the surface topography of Nitinol and Titanal after heat sterilization[6]. Kapilla, on the contrary, had found that clinical recycling combined with dry heat sterilization indeed alters the load deflection characteristics and surface topography[4,5].

There has been few comparative studies on the general properties as well as the influences of heat sterilization on Korean nickel titanium arch wires that has been in commmercial use since 1991. The purpose of this study was to investigate the changes in mechanical properties and surface topography of nickel titanium wires including a Korean product.

MATERIALS AND METHODS

(1) Materials

The materials used, as shown in Table 1, were 4 kinds of nickel titanium arch wires from different manufacturers. Rectangular wires of 0.016×0.022 inches were selected.

(2) Methods of sterilization

Two types of heat sterilization method approved by American Dental Association were used. Dentronix (Model DDS 5000) was utilized for dry heat sterilization, at the rate of 180℃ for 60 minutes, and Autoclave (Model 9041) for autoclave sterilization at the rate of 121℃, 15-20 psi for 30 minutes.

(3) Method of experimentation

The purpose of the experiment is to obtain, first of all, a comparative evaluation of the changes in the mechanical property through the use of tensile test, and second, a quantitative and qualitative evaluation of the changes in surface topography.

Twelve arch wires from each of the manufacturers were divided into three groups of four wires each. The first group of four was used as the control group (C), while the second group (dry heat group, D), and the third group (autoclave group, A) were subjected to sterilization.

a. Tensile test

With the Instron(Model 4466), 1000kg capacity of load cell was used. The focal distance was set at 40mm while the crosshead speed was at 5mm/min.

Out of the four wires in each of the three groups, three wires were put through the tensile test. The cross sectional area was determined by transporting the data through Series IX software, and three of the following variables were calculated.

① ultimate tensile strength (kg · f/mm^2)
② elongation ratio (%)
③ modulus of elasticity (kg · f/mm^2)

b. Experiments for surface topography

In order to evaluate the changes in the surface condition of the arch wires, scanning electron microscopy (JSM-840A JELO corp.) was used for observation. Also, to quantitatively evaluate the surface roughness,

Table 2. Summary of tensile properties before and after heat sterilliation (kg f/mm^2)

	Align		NiTi		Optimalloy		Sentalloy	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Ultimate tensile strength (kg · f/mm^2)								
Control	137.15	6.88	116.15	1.00	123.24	4.08	113.17	1.36
Autoclave	141.31	5.21	117.80	0.52	125.76	1.40	113.53	1.00
Dry heat	133.12	4.61	117.65	0.71	124.77	1.44	112.95	0.58
Enlongation rate (%)								
Control	26.17	1.18	18.72	1.10	23.73	1.61	22.75	0.20
Autoclave	25.81	1.45	18.40	0.64	23.76	0.54	22.89	0.29
Dry heat	25.93	0.74	18.28	0.45	23.77	1.44	22.56	0.62
Modulus of elasticity (kg · f/mm^2)								
Control	524.02	8.58	621.88	39.18	520.20	17.33	497.43	8.30
Autoclave	548.88	39.81	640.65	24.44	529.60	16.65	496.03	7.56
Dry heat	536.12	42.98	643.82	19.65	525.09	6.70	500.87	15.82

Table 3. The effect of heat sterilization on tensile properties of various nickel titanium wires tested statisctically by student t-test (n=3)

		Align		NiTi		Optimalloy		Sentalloy	
		p value	sig.	p value	sig.	p value	sig.	p value	sig.
Ultimate tensile strength	C vs A*	0.304	NS	0.063	NS	0.484	NS	0.503	NS
	C vs D**	0.590	NS	0.268	NS	0.448	NS	0.858	NS
Enlongation rate	C vs A	0.544	NS	0.440	NS	0.966	NS	0.651	NS
	C vs D	0.842	NS	0.655	NS	0.959	NS	0.635	NS
Modulus of elasticity	C vs A	0.322	NS	0.205	NS	0.071	NS	0.770	NS
	C vs D	0.100	NS	0.567	NS	0.600	NS	0.806	NS

(* control vs autoclave, ** control vs dry heat)

which influences the frictional characteristics of orthodontic arch wires, 3D profilogram (Optical dimensional metrology center) was used.

By this method, four separate regions of each specimen were tested, their surface roughness determined using the two variables of R_a(Roughness average) and R_q(Root mean square) for comparison.

RESULTS

1. Tensile properties

Neither method of heat sterilization had any effects on tensile properties of each nickel-titanium wire used in this experiment(Table 2, 3).

Fig 1. SEM findings (×500) of Align: It could be observed longitudinal indentation which seemed to be produced during drawing procedure (a). After dry heat sterilization (b), and autoclave (c), any changes of surface conditions were not observed.

Fig 2. SEM findings (×500) of NiTi: It could be observed longitudinal indentation which seemed to be produced during drawing procedure (a). After dry heat sterilization (b), and autoclave (c), any changes of surface conditions could not be noticeable.

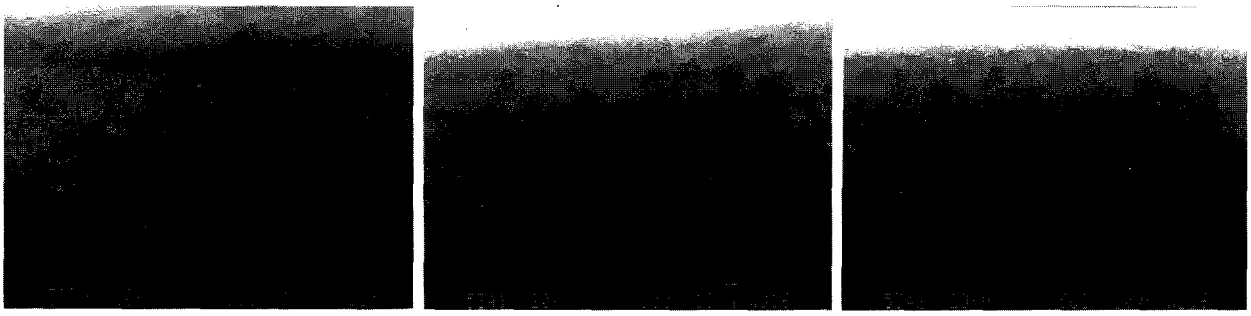

Fig 3. SEM findings (×500) of Optimalloy: No drawing trace was found in the control group, nor any indentation on the surface (a). No significant changes occurred in the surface condition following either heat sterilization procedures (b, c).

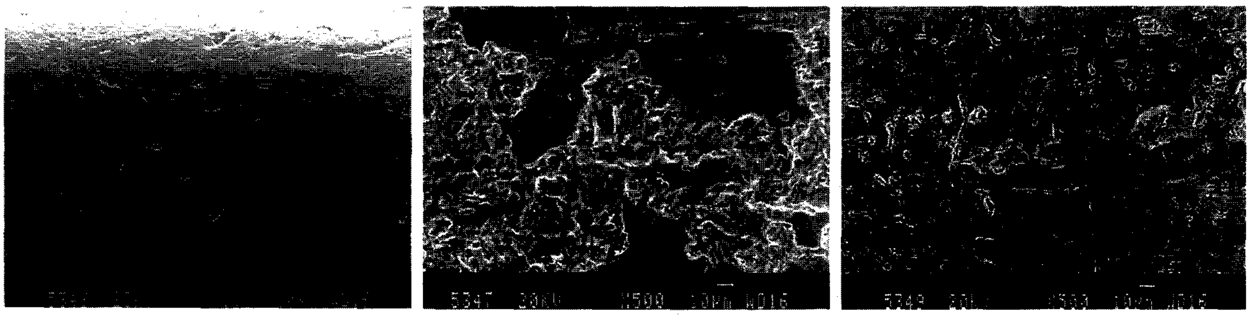

Fig 4. SEM findings (×500) of Sentalloy: In the control group, no drawing indentation was found but there was a distribution of small generalized pittings (a). Following each heat sterilization, the pittings became even more pronounce, with increase irregularities on the scoured surface (b, c).

Table 4. Surface roughness (R_a and R_q) in various nickel titanium wires before and after two kinds of heat sterilization method (unit : nm)

	Align		NiTi		Optimalloy		Sentalloy	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
R_a								
Control	459.8	28.3	738.2	106.4	117.8	36.3	446.6	7.7
Autoclave	406.2	45.1	701.6	37.0	105.5	11.7	694.3	88.5
Dry heat	420.0	20.4	894.3	95.1	1108	13.4	507.0	15.3
R_q								
Control	589.7	34.5	926.6	131.1	134.4	18.0	580.5	8.4
Autoclave	556.8	43.1	911.6	47.1	156.1	8.6	872.4	94.7
Dry heat	554.7	36.6	1110.0	107.0	148.2	17.1	650.6	18.0

Table 5. The effect of heat sterilization on surface roughness of various nickel titanium wires tested statistically by student t-test (n=4)

		Align		NiTi		Optimalloy		Sentalloy	
		p value	sig.	p value	sig.	p value	sig.	p value	sig.
R_a	C vs A[#]	0.061	NS	0.315	NS	0.251	NS	0.005	**
	C vs D[##]	0.050	NS	0.007	**	0.310	NS	0.002	**
R_q	C vs A	0.076	NS	0.438	NS	0.097	NS	0.004	**
	C vs D	0.197	NS	0.004	**	0.178	NS	0.002	**

([#] control vs autoclave, [##] control vs dry heat)

2. Surface topography

1) SEM findings

When the surface condition of the four products of the control groups that were not sterilized were viewed, a great deal of long axis indentations, that were probably caused by the drawing process, were apparent in the cases of Align and NiTi (Fig.1, 2). In both products, however, no significant changes occurred in the surface condition following either heat sterilization procedures. As for Optimalloy, no drawing trace was found in the control group, nor any indentation on the surface. And, again, no significant changes occurred in the surface condition following either heat sterilization procedures (Fig. 3). Although no drawing indentation was found in the Sentalloy control group, there was a distribution of small generalized pittings. And in the Sentalloy wires that were subject to sterilization, the pittings became even more pronounce, with increase irregularities on the scoured surface(Fig. 4).

2) Surface roughness

Only Sentalloy showed any change following each kind of heat sterilization procedures, with noticeable increase in the value of the R_a and the R_q. In NiTi there was increased surface roughness after the dry heat sterilization, while no significant change was observed in Align and Optimalloy in the dry heat group and the autoclave group(Table 4, 5).

DISCUSSION

1. Tensile Property

Segner, in his research comparing the mechanical property of nickel titanium wires manufactured by various companies, found a great deal of variation in the superelasticity among the 15 types of products he examined[9]. Even among just the nickel titanium wires, there were some differences in the mechanical property depending not only on the ingredient of the raw material, but also on the condition under which the product was handled at every step of the manufacturing process.

Among the products in the control group, Align showed the greatest tensile strength, followed by Optimalloy, and with NiTi and Sentalloy exemplifying the lowest value(Fig. 5, 6).

When it comes to the modulus of elasticity, NiTi showed the highest stiffness, followed by Align and Optimalloy, and with Sentalloy showing the lowest value (Fig 7).

None of the four products showed any change after heat sterilization procedures, which concurs with the results of the experiment by Mayhew where he observed tensile properties of two types products of nickel-titanium wires after heat sterilization[6].

This, however, differs slightly from the results obtained by Kapila, who put two types of nickel titanium wire in vivo through clinical recycling and dry heat sterilization, after which he determined the load deflection characteristics using 3 point bending test[4]. Although he ascertained that there was a small amount of change following the procedures, he left the clinical significance of the findings open to question.

In our research, we also found an increase in the modulus of elasticity in NiTi, Optimalloy and Sentalloy following dry heat sterilization, but the values were indeed small and without statistical significance.

It can be stated, then, there is no clinical significance of changes in mechanical properties brought about by heat sterilization.

2. Surface topography

1) SEM findings

In order to learn more about changes in surface topography, we used scanning electron microscopy to observe the results following each kind of heat sterilization. Just as there were some differences in the mechanical properties among the various products, there was a great deal of variance in the surface topographic condition among the four products. As shown in figure 1, 2, drawing indentations following the long axis remain in the Align and NiTi of the control group. No drawing indentations were found in the case of Sentalloy (Fig. 4), but a great amount of pitting was observed. Among the four products, Optimalloy had the smoothest surface topography with no drawing indentations or pitting visible (Fig. 3).

This resulted from the manufacturing procedure of the product, and it can be speculated that in the case of Align and NiTi that no special polishing was done after drawing, cutting and shape memory procedure. In the case of Sentalloy and Optimalloy, however, there was probably some polishing done in order to remove irregularities such as drawing indentation.

With the exception of Sentalloy, the remaining three products showed no observable changes in the surface topography following autoclave or dry heat sterilization. In Sentalloy, however, there was increased pitting in numerous areas of the surface. This probably arose from some treatment done during the manufacturing and polishing procedures.

2) Surface roughness

The quantitative value, the R_a(roughness average) and the R_q(roughness root mean square), of surface roughness was obtained through the use of profilo-meter.

Ra is usually used to express surface roughness value determined by mechanical means, and is the variable that can be applied in the easiest and the most stable manner. That is why it is used for the analysis of surface condition in general and as well as quality control during the manufacturing process. Rq, on the other hand, describes the optical finishing status of the

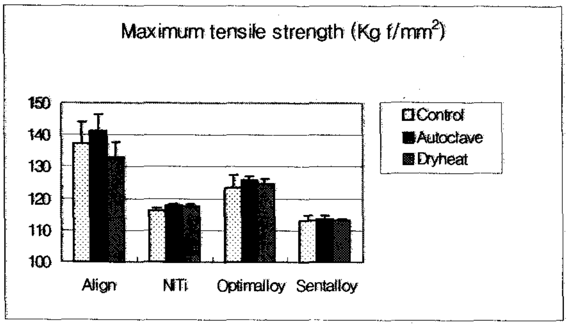

Fig 5. The maximum tensile strength of each nickel titanium wires did not changed significantly after two type of heat sterilization methods.

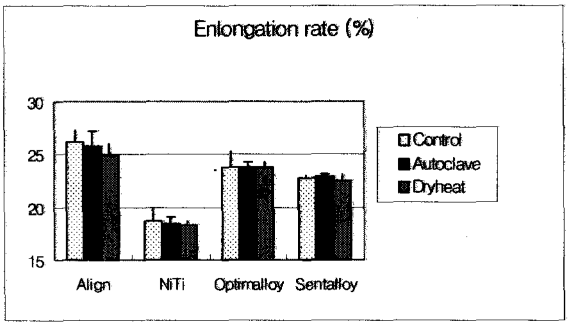

Fig 6. The elongation rate of each nickel titanium wires did not changed significantly after two type of heat sterilization methods.

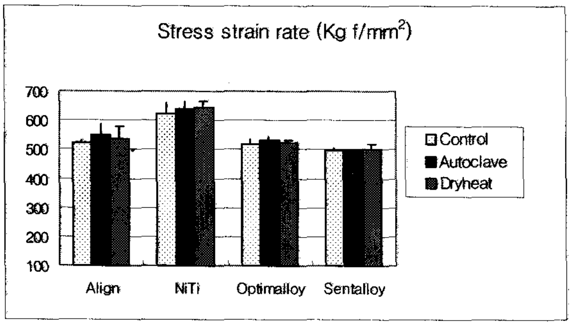

Fig 7. The stress strain rates of each nickel titanium wires tend to increase slightly except sentalloy but these changes after two types of heat sterilization methods are not significant statistically.

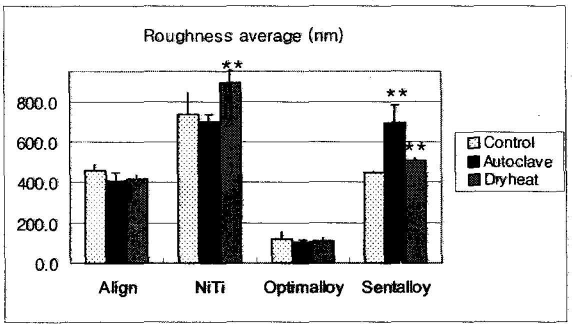

Fig 8. The surface roughness represented by R_a of NiTi is increased after dry heat sterilization and one of Sentalloy also increased after two types of sterilization methods. The surface roughness of Align and Optimalloy are not affected by heat sterilization.

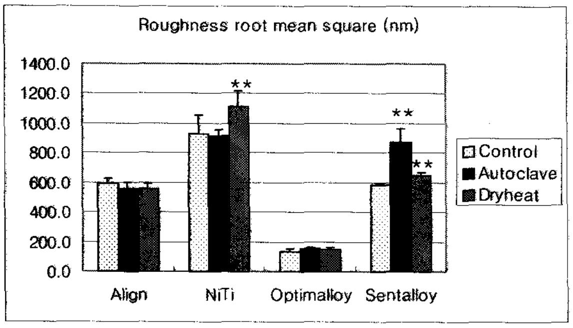

Fig 9. The surface roughness represented by R_q of NiTi is increased after dry heat sterilization and one of Sentalloy also increased after two types of sterilization methods.

surface, and serves as an important statistical data as it express the standard variation of the specimen surface. In our research, we found the same tendency in the values of the R_a and the R_q.

When the surface roughness value of the products in the control group were compared, NiTi had the highest rate of surface roughness, followed by Align and Sentalloy which had simillar values. The surface roughness value of Optimalloy was 1/4 that of Sentalloy and Align, and 1/7 of NiTi, thus exemplifying the lowest value of surface roughness (Fig. 8, 9). This agrees with the pattern observed through SEM.

In our research, a significant increase in the surface roughness was observed in NiTi after dry heat sterilization and Sentalloy after autoclave as well as dry heat sterilization.

Mayhew, in order to quantitatively evaluate surface topography after heat sterilization, used laser spectroscopy. According to this research, neither Nitinol nor Titanal showed any significant change after undergoing three types of heat sterilizations. The reason for the discrepancy in the results of our research and that of Mayhew, we believe, lies in the different methods used for the quantitative evaluation of surface topography[6].

While Mayhew used laser spectroscopy to measure the specular reflectivity of laser scattering, our research used the profilometer to obtain 3 dimensional measurement of up to 0.1nm, thus producing more meaningful results in quantifying changes in surface roughness.

Thayer, in his study, mentioned that cold work and heat treatments are important variables to be controlled during the manufacture of nitinol products[10]. It follows that there is a need to develop manufacturing procedure that maintains the stability in the various properties of nickel titanium wire following clinical recycling or heat sterilization.

CONCLUSION

1. Neither method of heat sterilization had any effects on tensile properties of the nickel-titanium wires used in this experiment.
2. Before heat sterilization, the surface smoothness was highest in Optimalloy, followed by Align and Sentalloy, with NiTi showing the lowest smoothness value.
3. In surface topography, Align and Optimalloy were not influenced by heat sterilization. NiTi, on the other hand, had increased roughness after dry heat sterilization and Sentalloy showed the same tendency after each of the two heat sterilization procedures.

REFERENCES

1. Buckthal JE, Mayhew MJ, Kusy RP, Crawford JJ. Survey of sterilization and disinfection procedures. J Clin Orthod 1986;20:759-65.
2. Edie JW, Andreasen GF, Zaytoun MP. Surface corrosion of nitinol and stainless steel under clinical condition. Angle Orthod 1981;51:319-324.
3. Harris EF, Newman SM, Nicholson JA. Nitinol arch wire in a simulated oral environment: Changes in mechanical properties. Am J Orthod 1988;93:508-13
4. Kapila S, Haugen JW, Watanabe LG. Load-deflection characteristics of nickel-titanium alloy wires after clinical recycling and dry heat sterilization. Am J Orthod 1992;102:120-6.
5. Kapila S, Reichhold GW, Anderson RS. Effect of clinical recycling on mechanical properties of nickel-titanium alloy wires. Am J Orthod 1991;100:428-435.
6. Mayhew MJ, Kusy RP. Effects of sterilization on the mechanical properties and the surface topography of nickel-titanium arch wires. Am J Orthod Dent Orthop 1988;93:232-6
7. Sarkar NK, Schwaninger B. The in vivo corrosion of nitiniol wire. J Dent Res 1980;59A:528
8. Schwaninger B, Sarkar NK, Foster BE. Effect of long-term immersion corrosion on the flexural properties of Nitinol. Am J Orthod 1982;82:45-9
9. Segner D, Ibe D. Properties of superelastic wires and their relevance to orthodontic treatment. Europ J Orthod 1995, 17:395-402.
10. Thayer, Bagby, Moore, and DeAngelis X-ray diffraction of nitinol orthodontic arch wires Am J Orthod Dent Orthop 1995;107;604-12

국문초록

열멸균과정이 nickel titanium호선의 기계적 성질과 표면상태에 미치는 영향

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본 연구의 목적은 수종의 nickel titanium 호선을 열멸균 처리 하였을 때 나타나는 물리적성질과 표면상태의 변화를 정량적으로 평가하고자 하는 것이다. 연구에 사용된 재료는 국산제품을 포함한 4종의 .016×.22 각형 nickel-titanium호선이며, 이들을 건열소독(180℃, 60분)군과 가압증기멸균(121℃, 15-20 psi, 30분)군, 그리고 아무런 처리를 하지 않은 대조군으로 구분하였다. 인정특성은 Inston (Model 4466, load cell capacity : 1000kg)을 이용하여 표점거리 40mm, cross head는 분당 5mm의 속도, 실온의 조건에서 평가하였으며 최대인장강도와 연신율, 그리고 탄성률을 측정 및 계산하였다. 표면상태는 주사전자현미경과 profilometer로써 평가하였고, t-test를 이용하여 통계적 분석을 하였다.

결과는 다음과 같다.

1. 실험에 사용된 nickel-titanium 호선들은 모두 두가지 열멸균 처리에 의해 인장특성에 있어서 유의할 만한 변화를 보이지 않았다/
2. 대조군에서 NiTi가 가장 높은 펴ㅛ면거칠기를 보였고, Align과 Sentalloy는 동일한 정도의 값을, Optimalloy는 가장 낮은 값을 지니고 있었다.
3. Align과 Optimalloy는 두가지 열멸균처리 후에 모두 유의할 만한 표면상태의 변화를 보이지 않았다. 그러나 NiTi는 건열소독후에 표면거칠기의 증가를 보였으며 Sentalloy는 두가지 열멸균후에 표면상태의 변화와 표면거칠기의 유위한 증가를 보였다.

주요단어 : 열멸균, 표면거칠기, 인장특성, nickel-titanium