

Comparison of transition temperature range and phase transformation behavior of nickel-titanium wires

Yu-Hyun Lee, DDS, PhD,^a Bum-Soon Lim, BA, PhD,^b Yong-Keun Lee, DDS, PhD,^c Cheol-We Kim, DDS, PhD,^d Seung-Hak Baek, DDS, PhD^c

Objective: The aim of this research was to evaluate the mechanical properties (MP) and degree of the phase transformation (PT) of martensitic (M-NiTi), austenitic (A-NiTi) and thermodynamic nickel-titanium wire (T-NiTi). **Methods:** The samples consisted of 0.016 × 0.022 inch M-NiTi (Nitinol Classic, NC), A-NiTi (Optimalloy, OPTI) and T-NiTi (Neo-Sentalloy, NEO). Differential scanning calorimetry (DSC), three-point bending test, X-ray diffraction (XRD), and microstructure examination were used. Statistical evaluation was undertaken using ANOVA test. **Results:** In DSC analysis, OPTI and NEO showed two peaks in the heating curves and one peak in the cooling curves. However, NC revealed one single broad and weak peak in the heating and cooling curves. Austenite finishing (A_f) temperatures were 19.7°C for OPTI, 24.6°C for NEO and 52.4°C for NC. In the three-point bending test, residual deflection was observed for NC, OPTI and NEO. The load ranges of NC and OPTI were broader and higher than NEO. XRD and microstructure analyses showed that OPTI and NEO had a mixture of martensite and austenite at temperatures below Martensite finishing (M_f). NEO and OPTI showed improved MP and PT behavior than NC. **Conclusions:** The mechanical and thermal behaviors of NiTi wire cannot be completely explained by the expected degree of PT because of complicated martensite variants and independent PT induced by heat and stress. (Korean J Orthod 2010;40(1):40-49)

Key words: Transition temperature range, Phase transformation, Ni-Ti wires

INTRODUCTION

Nickel-titanium (NiTi) wire has been widely used in the orthodontic field due to its shape memory effect,

super-elasticity and good biocompatibility.¹ There are three microstructural phases in the NiTi alloys ie. the austenitic phase, the high-temperature and low-stress form; the martensitic phase, the low-temperature and high-stress form; and the R-phase, an intermediate phase between martensite and austenite transformation.² The relative proportion of the austenitic and martensitic phases within transformation temperature range (TTR) seems to play an important role in the mechanical characteristics of NiTi wire.²⁻⁴

When NiTi wire is ligated to brackets on misaligned teeth in a temperature-fluctuant oral environment, slight thermal change can considerably modify the load level of NiTi wire.^{5,6} A discrepancy between transformation characteristics and related mechanical properties of NiTi wires has also been reported.⁷⁻¹⁰

^aPrivate Practice.

^bProfessor, ^cFormer Professor, ^dProfessor Emeritus, Department of Dental Biomaterials Science, Dental Research Institute, School of Dentistry, Seoul National University.

^eAssociate Professor, Department of Orthodontics, School of Dentistry, Seoul National University.

Corresponding author: **Seung-Hak Baek.**

Department of Orthodontics, School of Dentistry, Dental Research Institute, Seoul National University, 28, Yeongeon-dong, Jongno-gu, Seoul 110-768, Korea

+82 2 2072 3952; e-mail, drwhite@unitel.co.kr.

Received May 14, 2009; Last Revision October 19, 2009;

Accepted October 21, 2009.

DOI:10.4041/kjod.2010.40.1.40

Several commercially available NiTi wires are available such as the work-hardened martensitic type (M-NiTi), superelastic austenitic (A-NiTi) and thermodynamic (T-NiTi). Although M-NiTi wires can exhibit shape memory characteristics, their TTR does not seem to be clinically relevant for this property to be used for orthodontic treatment.¹¹ Burstone et al.¹² and Miura et al.¹³ introduced A-NiTi for superelasticity. More recently, T-NiTi wires have been developed for shape memory effect as well as superelasticity.¹⁴ However, these NiTi wires exhibit unexpected mechanical properties under stepwise temperature changes.^{10,15} Therefore, the purpose of this study was to evaluate the mechanical properties and degree of the phase transformation of commercially available M-, A- and T-NiTi wires under conditions of controlled temperature.

MATERIAL AND METHODS

Three types of 0.016 × 0.022 inch commercially available NiTi wires, M-NiTi (Nitinol Classic, 3M-Unitek, Monrovia, CA, USA; NC), A-NiTi (Optimalloy, Jinsung Medical, Seoul, Korea; OPTI), and T-NiTi (Neo Sentalloy, GAC, Tokyo, Japan; NEO) were used in this study (Table 1).

Differential scanning calorimetry (DSC) analysis

Transformation temperatures were measured using DSC 204 (Netzche, Germany). Specimens of each wire with the same length (4 mm) were sealed in an aluminum cell and placed into the measuring chamber, which was filled with argon gas. α -alumina was used as the reference material. The scanning temperature ranged from -20 to 100°C at 5°C/min. The measure-

ments were taken three times and were statistically analyzed using analysis of variance (ANOVA).

Three-point bending test

Three-point bending tests were performed using a universal testing machine (Instron 4465, UK) in a temperature-controlled water bath. The span length between the two supports was 14 mm. A bending load was applied to the wire specimen at a rate of 1.0 mm/minute to give a 2.0 mm deflection, and the load was subsequently removed at the same rate. The wire deflection was carried out at four different temperatures (0, 20, 37 and 60°C). The measurements were taken three times and were statistically analyzed by ANOVA.

Microstructure examination

The specimens were embedded in a self-curing resin and were electropolished for 6 - 7 minutes in a CH₃COOH and HClO₄ solution. The final surface was prepared by etching with a mixture of hydrofluoric acid (48% concentration), nitric acid (70% concentration), and deionized water in proportions of 1 : 4 : 10 by volume to reveal the grain boundary microstructure. Microstructure was examined using the metallographic microscope (IEM405, Zeiss, Germany).

X-ray diffraction (XRD) analysis

XRD was used to identify the crystal structure and relative portion of the austenite and martensite at various temperatures. An XRD Image Processor (DIP 2030, MAC science, Japan) was used with Cu-K α radiation at 40 KV and 80 mA for the low temperature measurements (-20, 5 and 20°C). In a pilot study, the

Table 1. Nickel-Titanium (NiTi) orthodontic wires used in this study

Species	Code	Brand name	Batch number	Manufacturer
M-NiTi	NC	Nitinol classic	W5084	3M Unitek, U.S.A.
A-NiTi	OPTI	Optimalloy	NO20805-016	Jinsung Meidcal, Korea
T-NiTi	NEO	Neo sentalloy	E589	Tomy International, Japan

M-NiTi means work-hardened martensitic type; A-NiTi, super-elastic austenitic type; T-NiTi, thermodynamic type.

XRD results at 0°C where the three-point bending test was performed could not be differentiated with that of the 5°C data. Therefore, the XRD test was performed at -20°C. The sampling stage was oscillated from 0 to 60°, 2θ in 0.01 degree steps to minimize the effect of the preferred orientation of wires. For the phase distribution analysis of martensite (002 peak) and austenite (110 peak) wires, FWHH (full width at half height) was compared. Powder XRD (D5005, Bruker, Germany) was used for the higher temperature measurements (37 and 60°C). The temperature was controlled by an electrothermic system and liquid nitrogen.

RESULTS

In the definitions of the transformation temperatures such as Ms, Mf, As, and Af, M means martensite; A, austenite; S, starting temperature; and f, finishing temperature, respectively.

DSC analysis

For NC (M-NiTi type), one single broad and weak peak was observed in the heating and cooling curves, respectively (Fig 1A). However, OPTI (A-NiTi type) and NEO (T-NiTi type) exhibited two endothermic peaks in the heating curves. It indicates that there may be an intermediate phase (R-phase: rhombohedral phase) during transformation. One exothermic peak (around 15 - 18°C) observed on the OPTI and NEO in the cooling curves could be considered as the reverse transformation from austenite phase to martensite phase (Fig 1B and C).

The TTRs for the three specimens were statistically different ($p < 0.05$, Table 2). Ms in the cooling process of NEO (T-NiTi type, 20.2°C) was higher than OPTI (A-NiTi type, 17.0°C). Various Af were found; 19.7°C for OPTI (A-NiTi type), 24.6°C for NEO (T-NiTi type) and 52.4°C for NC (M-NiTi type), respectively (Table 2).

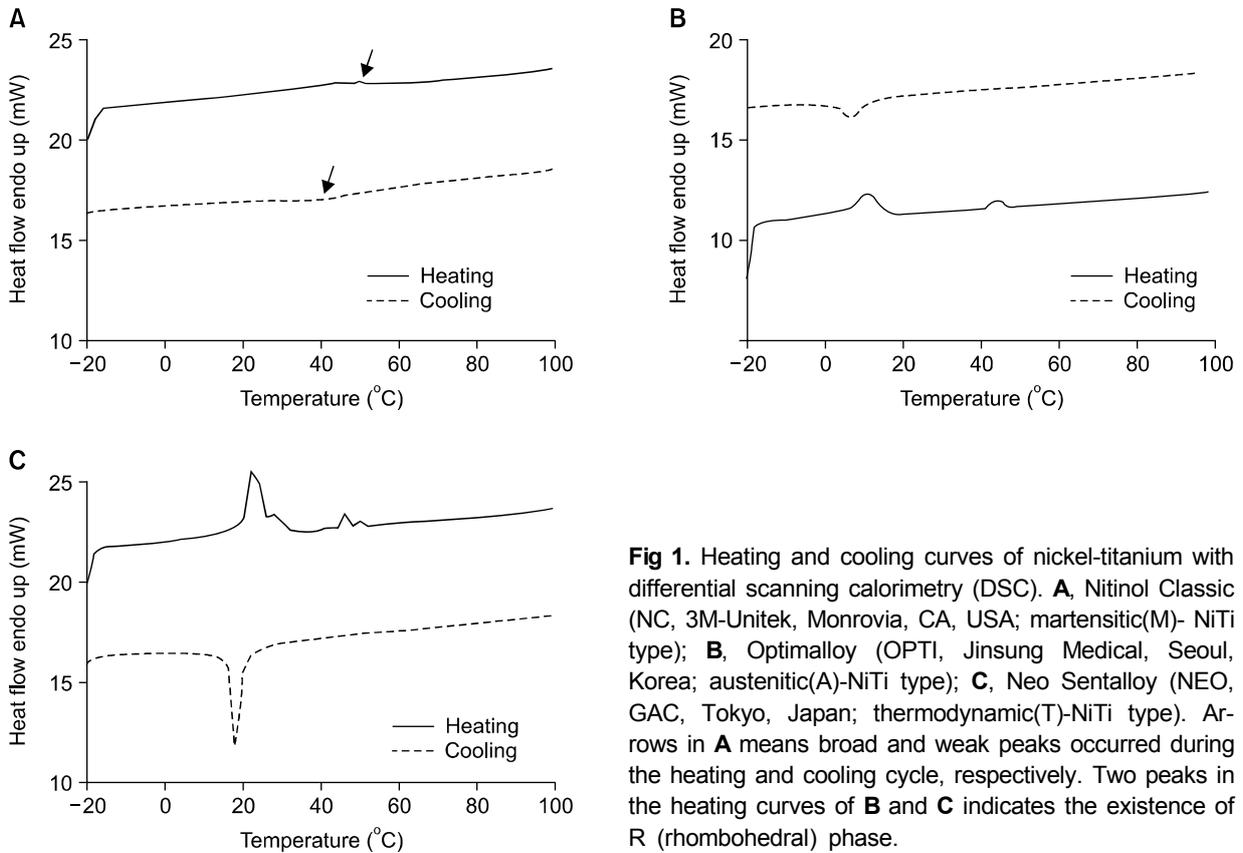


Fig 1. Heating and cooling curves of nickel-titanium with differential scanning calorimetry (DSC). **A**, Nitinol Classic (NC, 3M-Unitek, Monrovia, CA, USA; martensitic(M)- NiTi type); **B**, Optimalloy (OPTI, Jinsung Medical, Seoul, Korea; austenitic(A)-NiTi type); **C**, Neo Sentalloy (NEO, GAC, Tokyo, Japan; thermodynamic(T)-NiTi type). Arrows in **A** means broad and weak peaks occurred during the heating and cooling cycle, respectively. Two peaks in the heating curves of **B** and **C** indicates the existence of R (rhombohedral) phase.

Table 2. Comparison of the peak temperature during heating and cooling curves according to differential scanning calorimetry (DSC) analysis

Species	Code	Heating						Cooling					
		A _s (°C)	Peak (°C)	A _f (°C)	TTR* (°C)	ΔH (mJ)	ΔQ (J/g)	M _s (°C)	Peak (°C)	M _f (°C)	TTR* (°C)	ΔH (mJ)	ΔQ (J/g)
M-NiTi	NC	(48.5)	(50.5)	(52.4)	(3.9)	(1.8)	(41.2)	(55.4)	(40.1)	(8.3)	(47.1)	(-2.4)	(-53.9)
A-NiTi	OPTI	10.5	15.3	19.7	9.2	3.4	76.0	17.0	11.3	6.0	11.0	-2.9	-66.7
T-NiTi	NEO	20.6	23.0	28.0	7.4	5.8	137.5	20.2	18.1	16.0	4.2	-4.5	-106.0

ANOVA test was done. *Means $p < 0.05$; M, martensite; A, austenite; S, starting temperature; f, finishing temperature; TTR, transformation temperature range; ΔH, transition enthalpy; ΔQ, transition energy; M-NiTi, work-hardened martensitic type; A-NiTi, super-elastic austenitic one T-NiTi, thermodynamic one. (): not in the graphs, but was pitched with peak-picking function.

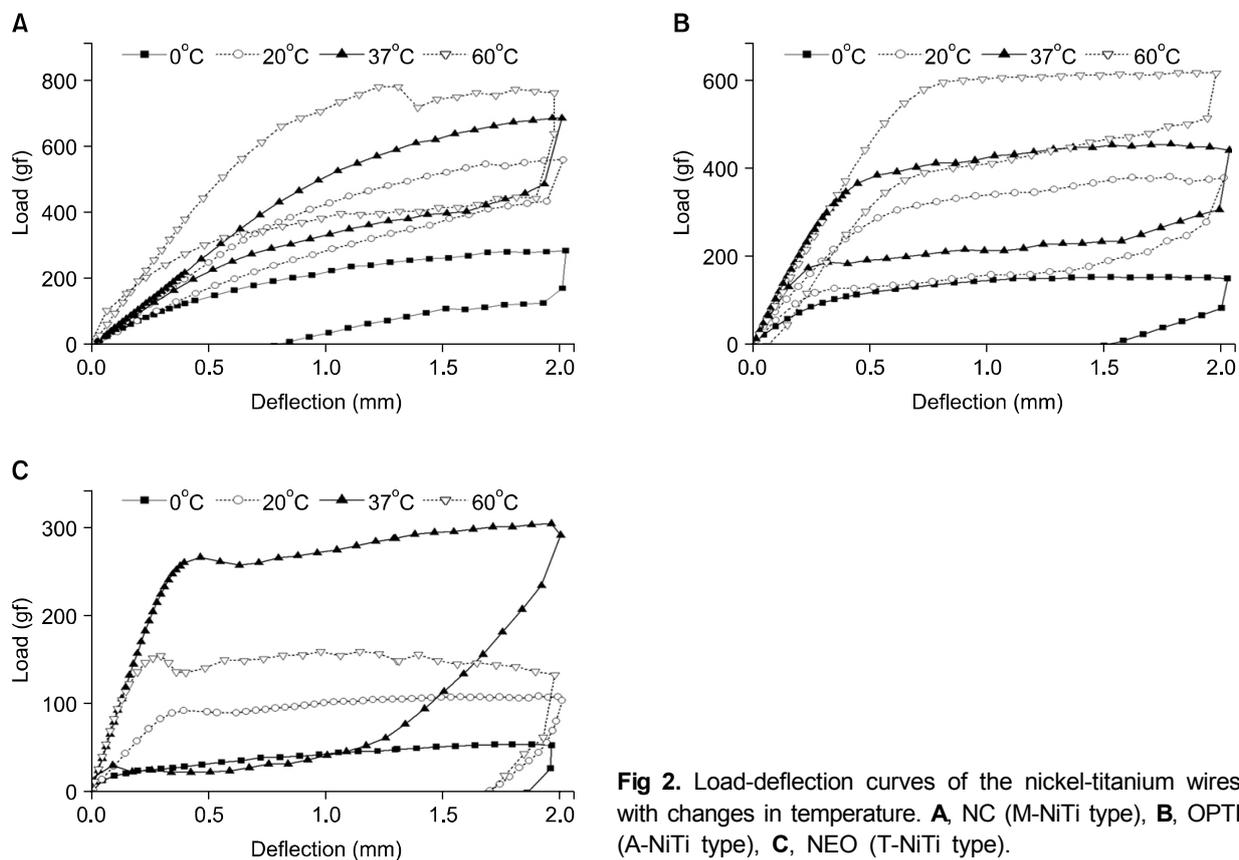


Fig 2. Load-deflection curves of the nickel-titanium wires with changes in temperature. **A**, NC (M-NiTi type), **B**, OPTI (A-NiTi type), **C**, NEO (T-NiTi type).

Three-point bending test

In NC (M-NiTi type), the superelasticity loop appeared at 60°C (Fig 2A and Table 3). Total recovery ($\delta = 0$) was observed at 20°C and 37°C (Table 3),

where the superelasticity loop did not appear (Fig 2A). Partial recovery at 0°C ($\delta = 0.8 \pm 0.02$, Table 3) might be an expression of the plastic deformation in the martensite phase.

In OPTI (A-NiTi type), total recovery was observed

Table 3. Comparison of the mechanical properties according to a function of temperature

Species	Code	Temp. (°C)	α load (gf)	β load (gf)	γ load (gf)	δ load (gf)	σ deflection (mm)
M-NiTi	NC	0	79.8 ± 5.05 [†]	300.5 ± 10.33 [†]	121.9 ± 24.70 [†]	-	0.8 ± 0.02
		20	365.6 ± 20.86 [†]	473.3 ± 30.26 [†]	356.6 ± 14.69 [†]	187.0 ± 8.40 [†]	0
		37	318.1 ± 6.43 [†]	453.0 ± 11.35 [†]	234.2 ± 22.21	115.0 ± 2.41	0
		60	429.7 ± 7.10	586.7 ± 23.00	353.1 ± 27.53	252.3 ± 18.39	0
A-NiTi	OPTI	0	110.2 ± 5.44	144.1 ± 6.72	-	-	1.6 ± 0.04
		20	308.8 ± 5.44	378.9 ± 8.94	167.6 ± 9.19	133.9 ± 8.60	0
		37	350.3 ± 28.69	416.0 ± 21.87	218.6 ± 13.37	160.7 ± 20.02	0
		60*	588.2 ± 2.46	596.5 ± 8.45	479.2 ± 12.09	351.2 ± 22.79	0
T-NiTi	NEO	0	22.7 ± 3.21	56.3 ± 2.82	-	-	1.9 ± 0.01
		20	98.8 ± 5.95	112.0 ± 4.86	-	-	1.7 ± 0.01
		37	254.6 ± 2.07	299.2 ± 4.15	42.9 ± 4.44	29.2 ± 2.64	0
		60	124.2 ± 11.80	136.5 ± 11.84	-	-	1.7 ± 0.03

ANOVA test was done. * Means $p < 0.01$. The four force change points and residual deflection were measured. α , Martensite transformation start point; β , martensite transformation finish point; γ , austenite re-transformation start point; δ , austenite re-transformation finish point; σ , residual deflection value at load reached zero in the unloading; M-NiTi, work-hardened martensitic type; A-NiTi, super-elastic austenitic type; T-NiTi, thermodynamic type; -, not-measurable parameters; [†] No discernible plateau.

except at 0°C (Fig 2B and Table 3). Since A_f of OPTI is 19.7°C (Table 2), the phases at 20, 37 and 60°C may be austenite. The position of the hysteresis loop at 60°C (Fig 2B) indicates that the load increased according to the increase in temperature ($p < 0.01$, Table 3). The residual deflection at 0°C ($\delta = 0.16 \pm 0.04$, Table 3) might imply the existence of deformation in the martensite structure.

NEO (T-NiTi type) exhibited superelasticity behavior only at 37°C (Fig 2C and Table 3). Since 37°C falls above the A_f of NEO (24.6°C, Table 2), the phase at 37°C may be austenite and the mechanical behavior is associated with formation of stress-induced martensite upon loading and by reverse transformation upon unloading. A partial recovery at 0°C and 20°C ($\delta = 1.9 \pm 0.01$, $\delta = 1.7 \pm 0.01$, respectively, Table 3) indicates that the residual stress remained within the wire and that the reverse transformation did not occur completely during unloading. A residual deflection at 60°C was also observed ($\delta = 1.7 \pm 0.03$, Table 3).

Microstructure examination

In NC (M-NiTi type), a wavy pattern like striated band was not identified, but a deformation line made by a work-hardening process was observed (Fig 3A). The stabilized work-hardened martensite plate, which might not undergo total retransformation, was observed in NC (M-NiTi type).

In OPTI (A-NiTi type), a wavy pattern like striated band was obvious (Fig 3B). NEO (T-NiTi type) showed evenly dispersed non-metallic inclusions and holes in the NiTi matrix (Fig 3C). Both OPTI (A-NiTi type) and NEO (T-NiTi type) showed mixed structures with a martensite plate in an austenite matrix (Fig 3B and C).

XDR analysis

For NC (M-NiTi type), the peak change could not be clearly analyzed due to the eccentric preferred orientation produced by work-hardening (Fig 4A).

The mixed pattern of a strong peak for austenite (PA, 110) and a weak peak for martensite (PM, 002)

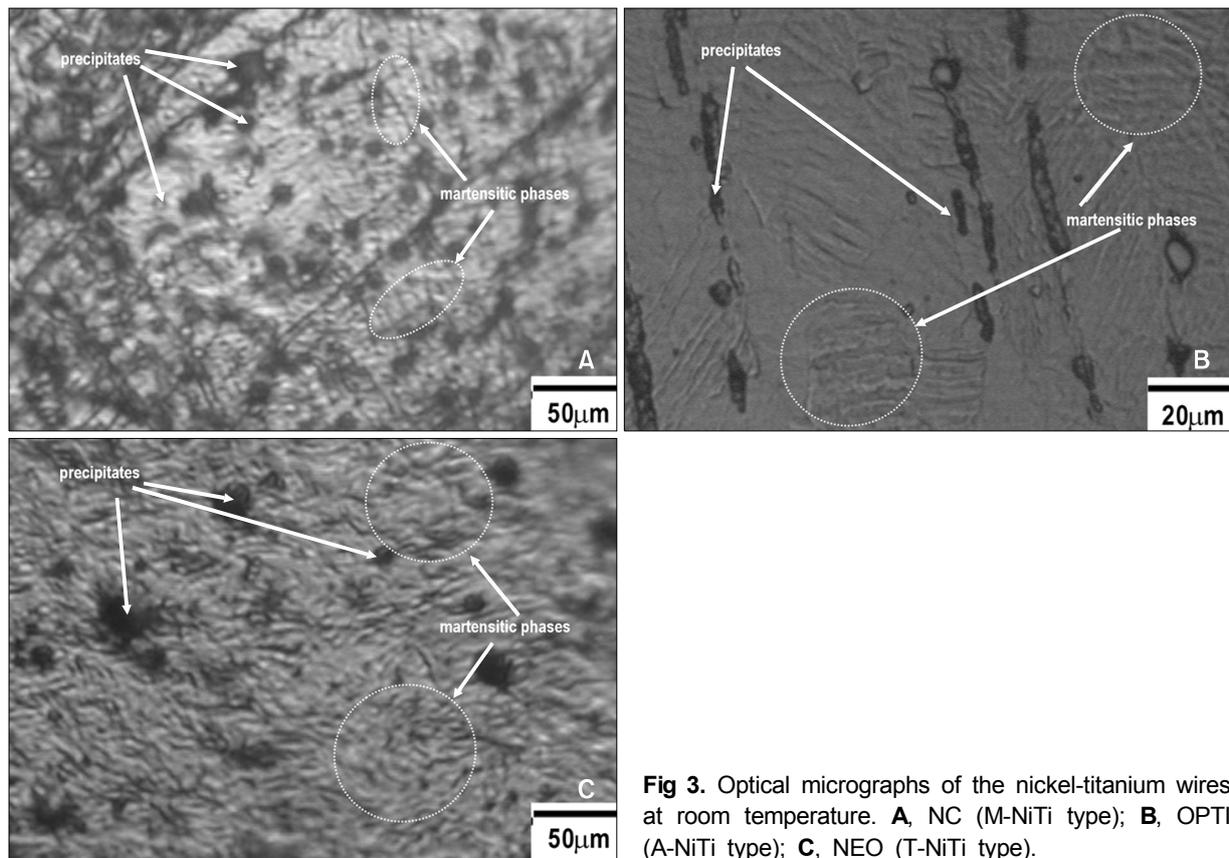


Fig 3. Optical micrographs of the nickel-titanium wires at room temperature. **A**, NC (M-NiTi type); **B**, OPTI (A-NiTi type); **C**, NEO (T-NiTi type).

was observed in OPTI (A-NiTi type, Fig 4B). The PM (002) did not become obvious when the temperature decreased to -20°C (Fig 4B).

In NEO (T-NiTi type), there was a mixed pattern of strong PA (110) and mild PM (002) at 5°C (Fig 4C). However, the finding that relatively stronger PM (002) and weaker PA (110) existed at -20°C than at 5 and 25°C (Fig 4C) indicates that even at a temperature below M_f , T-NiTi wire appeared to have a two-phase structure, not a single crystal structure of martensite. The PM (002) gradually disappeared when the temperature was increased above 25°C (Fig 4C).

The peak width is an indication for degree of cold work. Compared with full width at half height (FWHH) of the PA (110) peak, NC (M-NiTi type) exhibited a wider peak (more cold work) than either NEO (T-NiTi type) or OPTI (A-NiTi type) (Fig 5).

DISCUSSION

DSC and XRD analyses can provide useful information about the phase transformation and microstructure which are related with the mechanical properties of the NiTi wires.^{11,15-17} Since the transformation temperature of NiTi wires, A_f and M_s , are critical factors in their transformation behavior,¹⁸ it is important to choose an alloy with the correct A_f (for example, less than 37°C) to be clinically relevant. In this study, A_f of NC (M-NiTi type, 52.4°C , Table 2) determined by DSC analysis seems to be too high for clinical use. Since the heating DSC curves suggest that NEO (T-NiTi type) and OPTI (A-NiTi type) are completely austenitic in an oral environment (Fig 1), NEO and OPTI could be considered to act as superelastic wires.

Since the two endothermic peaks in the heating curves of OPTI and NEO (Fig 1) means an existence of R phase, differences in the DSC curves and XRD

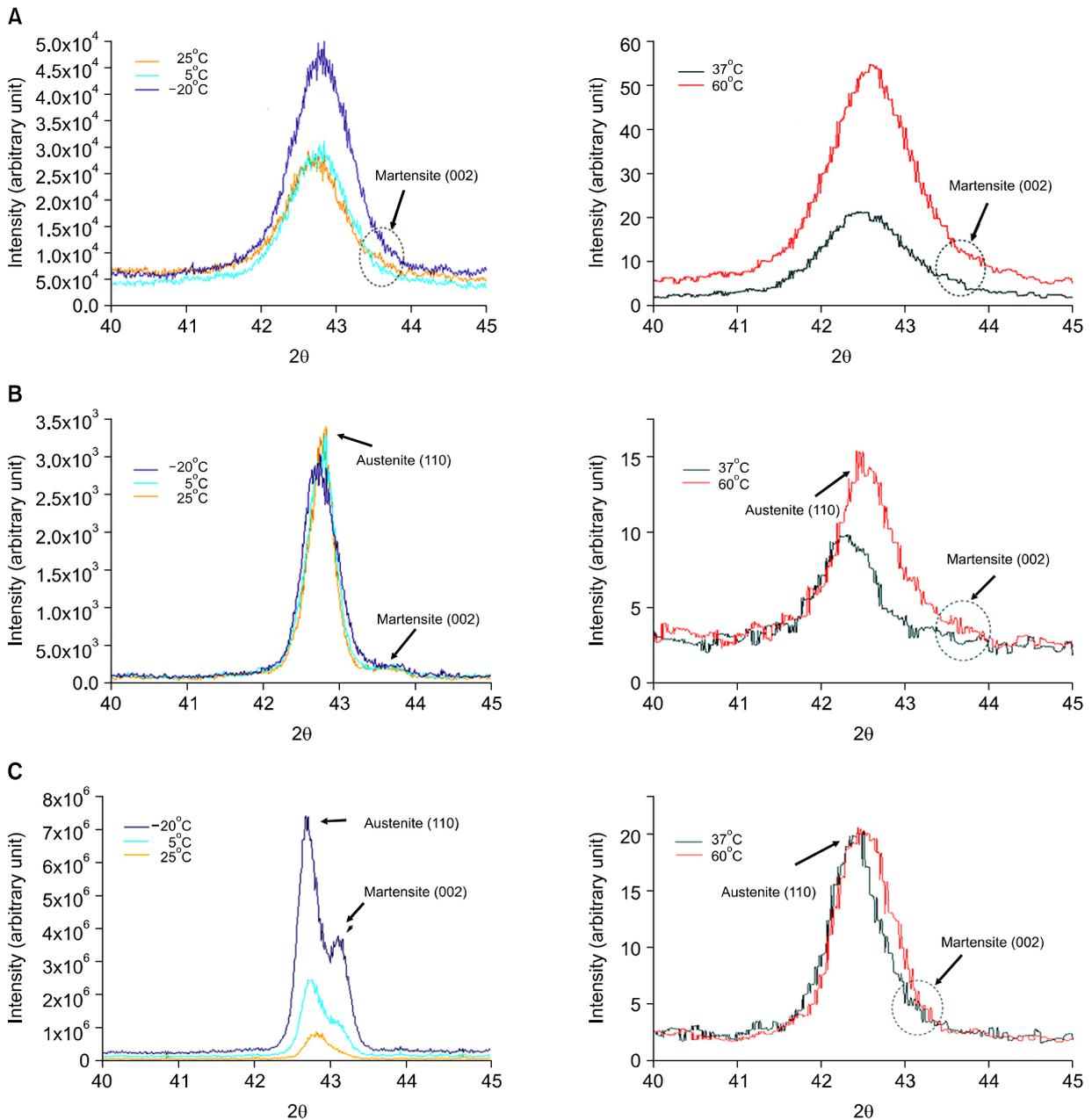


Fig 4. X-ray diffraction patterns of the nickel-titanium wires at various temperatures. **A,** NC (M-NiTi type); **B,** OPTI (A-NiTi type); **C,** NEO (T-NiTi type).

analyses among the three types of NiTi wires might be explained by the influence of the interposition of the R-phase in NEO and OPTI wires (Figs 1, 4 and 5 and Table 2).

The deformation temperatures are divided into four temperature regions; $T < M_s$, $M_s < T < A_f$, $A_f < T < T_d$ and $T_d < T$ (where T_d represents the critical

temperature where the plastic deformation by dislocation motion starts). The common features of the curves in Fig 2 are the presence of a residual deflection after unloading at 0°C ($T < M_s$), and the perfect recovery of the deflection at 37°C in all samples and 60°C ($T > M_s$) in OPTI (A-NiTi type) and NC (M-NiTi type). At 60°C , martensite re-transformation

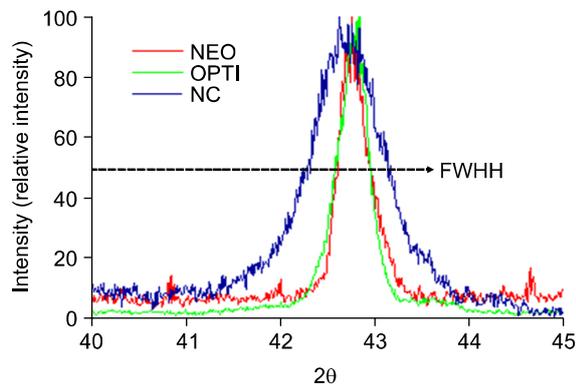


Fig 5. X-ray diffraction patterns of the nickel-titanium wires at room temperature. NC, M-NiTi type; OPTI, A-NiTi type; NEO, T-NiTi type. FWHH means full width at half height.

appears to be inhibited by the plastic deformation induced by dislocation in NEO (T-NiTi type) (Fig 2 and Tables 2 and 3).

At the temperatures below M_f , the martensite crystal is attained by continuous growth of the martensite plates and the nucleation of new martensite plates.² When the specimen is heated, the martensite plates revert completely to the parent phase and to the original lattice orientation.^{2,5} Another mechanism for achieving crystallographic reversibility is with the use of the stress-induced martensite phenomena.¹⁹ The mechanical behavior of OPTI (A-NiTi type) at room temperature (Fig 1B) is associated with the formation of stress-induced martensite upon loading and by reverse transformation on unloading. In general, the martensite is induced when $T < M_s$ or the boundaries between the martensites or internal twin boundaries begin to move. As the environmental temperature is elevated, the critical load of stress-induced martensite transformation is also increased. This is because the parent phase is more stable at high environmental temperatures. There is a negative linear relationship between the stress and the temperature with respect to the induction of martensite; a decrease in temperature is equivalent to an increase in stress.^{20,21} According to this relationship, the critical stress to induce martensite in a low A_f wire is higher than that in a high A_f wire. Therefore, the load, α , in Ni-Ti wire with a low A_f (OPTI, A-NiTi type, 19.7°C, Table 2) would be relatively high ($p <$

0.01, Table 3).

The mean mouth temperature throughout a 24-hour period is known to be around 35°C with variation according to time and location.²¹⁻²³ Since a transient change in the oral temperature associated with ingestion of cold/hot food can lead to marked changes in the phase transformation of NiTi wires, difference in force magnitude of the NiTi wires can cause irreversible tissue damage, extensive hyalinization of the periodontal ligament and root resorption or retardation of tooth movement.^{16,24} Therefore, the mechanical properties of these wires need to be measured in clinical temperature ranges.^{5,25-34} The results of this study clearly indicate that the mechanical properties of NiTi wires are substantially affected by temperature changes (Table 3). When the temperature was varied between 0 and 60°C, the load ranged between 22.7 and 124.2 gf for NEO (T-NiTi type), between 110.7 and 588.2 gf for OPTI (A-NiTi type), and between 79.8 and 429.7 gf for NC (M-NiTi type) (Table 3). The broad force range according to temperature change in OPTI (A-NiTi type) and NC (M-NiTi type) compared with NEO (M-NiTi type) (Table 3) might not guarantee a light and constant force for effective and efficient tooth movement.

Although the results of this study may explain the reason why commercially available M-, A- and T-NiTi wires exhibited complicated and unexpected mechanical properties according to temperature change, the details of the transformation behavior and mechanical properties of NiTi wires cannot be fully described. Therefore, further studies concerning the relationship among microstructure, phase transformation behavior and mechanical properties of NiTi wires will be needed.

CONCLUSION

1. NEO and OPTI showed better mechanical properties and phase transformation behavior than NC.
2. The mechanical and thermal behaviors of NiTi wire cannot be completely explained by the expected phase distribution and phase ratio from TTR because NiTi alloy has complicated martensite variants and independent phase transformation induced by

heat and stress.

- 국문초록 -

니켈-타이타늄 호선의 상전이 온도 범위와 상전이 행동 비교

이유현^a · 임범순^b · 이용근^c · 김철위^d · 백승학^e

본 연구의 목적은 martensitic (M-NiTi), austenitic (A-NiTi) 및 thermodynamic nickel-titanium wire (T-NiTi)의 물리적 특징과 상전이 정도를 평가하는 것이다. 재료는 0.016 × 0.022 inch의 M-NiTi (Nitinol Classic, NC), A-NiTi (Optimalloy, OPTI)와 T-NiTi (Neo-Sentalloy, NEO)이었으며, differential scanning calorimetry (DSC), 3점굽힘실험, X-ray diffraction (XRD), 미세구조 분석을 시행하였으며, ANOVA test로 통계 처리하였다. DSC분석 결과 OPTI와 NEO는 heating curve에서 2개의 peak, cooling curve에서 1개의 peak를 보였고, NC는 heating과 cooling curve에서 1개의 넓고 약한 peak를 보였다. Austenite finishing (A_f) 온도는 OPTI 19.7°C, NEO 24.6°C, NC 52.4°C였다. 3점굽힘실험 결과 NC, OPTI, NEO 모두 residual deflection을 보였으며, NC와 OPTI의 load range가 NEO보다 컸다. XRD와 미세구조 분석결과 OPTI와 NEO는 Martensite finishing (M_f)에서 martensite와 austenite가 섞여 있음이 관찰되었다. NEO와 OPTI는 NC보다 개선된 물리적 특징과 상전이 행태를 보였다. NiTi 호선의 물리적, 온도에 따른 행태는 예상되었던 상전이 정도에 의해 완벽하게 설명되지 않았으며, 그 이유는 복잡한 martensite variants의 존재와 열과 stress에 의해 유도된 독립적인 상전이에 기인한다고 생각된다.

주요 단어: Transition temperature range, Phase transformation, Ni-Ti wires

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