Formation Mechanism of Tempering-Induced Martensite in Ti–10Mo–7Al Alloy

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The formation mechanism of α''-martensite (α'' Mt) induced by tempering at 450–550°C for a short time was investigated using Ti–10Mo–7Al alloy. The solution treated and quenched (STQ) sample was composed of β phase and a small amount of α'' Mt, and a large amount of α'' Mt was generated by rapid tempering at 550°C for 3 s using a salt bath. However, α'' Mt was completely transformed into a single β phase by aging at 200°C for 3 min. Reversibility was observed between the α'' Mt transformation and the β reverse transformation. In-situ high-temperature X-ray diffraction measurements revealed that α'' Mt → β reverse transformation occurred at 200°C and that a thermally activated α'' iso was generated at 450°C due to the slow heating rate. In-situ optical microscopic observation of STQ sample with rapid lamp heating revealed that α'' Mt was formed during heating process. However, α'' Mt did not generate under following conditions; that is, a slow heating rate, thin sample plate, and a small temperature difference until tempering by preheating. On the other hand, rapid tempering using thick plate from liquid nitrogen (−196°C) to 250°C was performed to ensure a sufficient temperature difference, but α'' Mt was not generated at all.

From the cross-sectional observation of the STQ plate, it was found that α'' Mt was hardly formed on the surface of the sample, but was formed abundantly inside the sample. On the other hand, in the rapidly tempered plate, a large amount of α'' Mt was distributed in the surface layer than inside sample. These results suggest that the thermal compressive stress induced by rapid heat treatment contributes to the formation of α'' Mt.

1. Introduction

The α'' phase of β-type Ti alloys has an orthorhombic structure (Ccmcm)1,2 and can be classified into three types that vary in microstructure, size, and formation mechanism. The phase with the smallest size (~3 nm) is called nanodomain3–6 or O'' phase7–9 and has a very fine, modulated structure distributed uniformly in the β matrix of solution-treated and quenched (STQ) samples. This structure is [-110] shuffled in every other layer of the (110) stacked β-phase, and the axial ratio of the lattice constants, i.e., $b_\alpha''/a_\alpha'' = \sqrt{2}$, establishes a coherent relationship between the α'' and β phases. However, the α'' phase formation mechanism remains unclear. Nii et al.3 proposed a model in which impurity oxygen atoms in the Ti alloy caused lattice shear displacement and [-110] shuffling in the β phase. However, Zheng et al. reported that oxygen atoms are not necessary for the formation of the O'' phase.7

The second type of α'' phase, commonly referred to as α'' iso,8,9 is a needle-like product a few hundred nanometers in size, which forms during isothermal aging at relatively high temperatures (approximately 400°C). α'' iso is a diffusion transformation product generated and grown by thermal activation10,11 and is distinctly different from martensite. The structure of α'' iso varies with alloy composition, aging temperature, and aging time; the axial ratio $b_\alpha''/a_\alpha''$ of this phase has a value of $\sqrt{2} < b_\alpha''/a_\alpha'' < \sqrt{3}$. Without considering the composition, $b_\alpha''/a_\alpha'' = \sqrt{2}$ and $\sqrt{3}$ correspond to the O' and hexagonal close-packed α phases, respectively. The α'' iso compositional change is almost undetectable in low-temperature aging or in the early stage of higher-temperature aging10,12 with further aging, the concentration of β-stabilizing decreases, and the structure eventually shifts to the α phase.6 The α'' iso phase is produced rapidly and leads to significant hardening after a few seconds to a few minutes of higher-temperature aging11,12. During α'' iso formation, specific α'' iso variants are preferentially formed in response to slight strains in the matrix phase, resulting in a peculiar phenomenon, in which the sample shape and surface change significantly10,13–15. For example, a thin STQ plate bent into a U-shape at room temperature will continue to bend with increasing temperature. The peculiar phenomena associated with tempering that we have reported for Ti–(4Fe,10Mo,35Nb)–7Al are all due to α'' iso formation.13–15

The third phase is α'' martensite (α'' Mt), which is formed by several non-diffusion processes, such as STQ,16 sub-zero processing,17 and work processing;15 herein, we refer to these three products as α'' Mt, α'' iso, and α'' Mt, respectively. The size of the α'' Mt grains formed depends on the initial β grain size. This phase was identified earlier than the other two18 because it is a coarse, needle-like product up to several hundred micrometers in size and is easily observed by optical microscopy (OM). The O' and α'' iso phases have been reported since 2013,19 because of their extremely small size, these forms can only be observed under a transmission electron microscope.

Recently, an interesting α'' Mt formation was reported in Ti–15V–7Al alloy.17 The microstructure of this STQ alloy was mainly composed of residual β-phase with some α'' Mt near the grain boundaries. However, when the STQ sample was rapidly heated to 550°C–3 s in a salt bath and then rapidly cooled, coarse α'' Mt without compositional change (hereafter
denoted $\alpha''_{\text{Mf}}$) was formed over the entire specimen surface. Subsequent aging at 200°C resulted in a reverse transformation to a single $\beta$ phase, but tempering at 550°C for 3 s again reversibly produced $\alpha''_{\text{Mf}}$.

However, when the sample was quenched after being held at 550°C for more than 3 min, $\alpha''_{\text{Mf}}$ did not form. Instead, fine $\alpha''_{\text{iso}}$ grains with dilute vanadium precipitates were distributed in the $\beta$ matrix. Whether $\alpha''_{\text{Mf}}$ was formed during heating or during cooling after tempering is unclear at present. In the former scenario, martensite was produced using a non-consumption tungsten arc melting furnace and melted at 10.11Mo.

A 600 g alloy ingot was prepared using a non-consumption tungsten arc melting furnace and melted in a vacuum ($<1 \times 10^{-4}$ Torr) using characteristic X-ray CuK$\alpha$ in the range of $2\theta = 30^\circ$–$60^\circ$. The temperature was increased at a rate of 10°C/min and held at 200°C and 450°C for the measurements. To minimize the effect of the preferred orientation caused by the coarse crystals, a sample was cut from the ingot and a rotation stage was used. For the in-situ observation of rapid heating, a mirror-finished STQ sample (approximately $5 \times 3 \times 1 \text{mm}^3$) was rapidly heated to approximately 550°C at a rate of approximately 100°C/s using a condensing lamp in a vacuum ($<1 \times 10^{-4}$ Torr), and the microstructural changes were recorded. The thermal conductivity and specific heat were measured by the laser flash method at room temperature using an 8 mm$^3 \times 1 \text{mm}^3$ STQ sample. Young’s modulus was measured using the free resonance method\(^\text{(10)}\) at room temperature using an STQ sample (approximately $40 \times 3 \times 1 \text{mm}^3$). To investigate the work-induced $\alpha''_{\text{Md}}$ transformation stress, tensile specimens of the geometry shown in Fig. 9 were prepared; after STQ treatment, tensile tests were conducted in air at room temperature, 150°C, and 200°C until fracture at an initial strain rate of $8.33 \times 10^{-4}$ s$^{-1}$.

3. Results and Discussion

3.1 Formation and disappearance of $\alpha''_{\text{Mf}}$ during heat treatment

Figure 1 shows the OM microstructures of the 10MoA alloy after heat treatment: (a) STQ; (b) tempering at 550°C for 3 s using a salt bath; and (c) aging at 200°C for 3 min. The same specimen was used for observations (a) through (c). After heat treatment, the specimens were slightly mechanically and electrically polished and then chemically etched, and the same areas were observed. The STQ sample showed a $\beta + (\alpha''_{\text{Mf}})$ microstructure, but after tempering at 550°C for 3 s, coarse $\alpha''_{\text{Mf}}$ was formed over the entire surface. According to XRD measurements, the sample is composed of $\beta$ phase and $\alpha''_{\text{Mf}}$, and the lattice constants are $a_\beta = 0.326$ nm and $a_{\alpha''} = 0.305$ nm, $b_{\alpha''} = 0.485$ nm, $c_{\alpha''} = 0.467$ nm. $b_{\alpha''}/$
After aging at 200°C, the α' Mt disappeared, and a single phase was formed. To investigate the reversibility of the transformation, the sample was treated again at 550°C for 3 s (not shown in the figure). The entire surface of the sample became covered with α' Mt, which underwent reverse transformation after treatment at 200°C for 3 min. The above reversible microstructural change by heat treatment was similar to that of Ti-15V-7Al.17) Figure 2 shows the results of the in-situ XRD measurements at high temperature. The STQ sample structure at room temperature was α + (α' Mt); the α-phase lattice parameter is a = 0.326 nm. A reliable value for the lattice constant of α' Mt could not be determined because of the small amount produced. When the temperature was raised to 200°C, the profile approached that of the α single phase. When compared with the ex-situ, room temperature XRD profile11) of the sample tempered at 200°C for 5 min, the peaks measured at 200°C are very diffuse. This result could be an effect of the higher measurement temperature, which would increase the crystal lattice vibration and therefore the peak widths; it is also possible that a fine α' iso had been generated.10) When the temperature was further increased to 450°C, a single phase of α' iso (a' = 0.304 nm, b' = 0.488 nm, c' = 0.466 nm, b'/a' = 1.61) was obtained. This result is in good agreement with the transformation behavior expected from the TTT diagram10) proposed by Tahara et al. for Ti-3Mo-6Sn-6Al (mol%) alloys. When we compared the structure of α' iso with that of α' Mt produced by salt bath tempering at 550°C for 3 s, we found that the axial ratio of α' iso was larger and closer to the structure of the α phase.

To investigate whether α' Mt associated with tempering is formed during heating or during subsequent quenching, in-situ observations were conducted using rapid ramp heating, as shown in Fig. 3. An STQ plate of approximately 5 × 3 × 1 mm³ was polished to a mirror finish by mechanical and electrolytic polishing. A thermocouple was attached between the platinum stage and the bottom of the specimen, as shown in the schematic diagram; because focused heating was performed from the top of the specimen and from the bottom of the stage, the temperature immediately after the start of irradiation was higher on the top of the specimen than the actual measurement. Although a small amount of α' Mt was observed in the initial microstructure, this α' Mt disappeared after 1 s of heating, and the microstructure became flat. However, after 2 s, needle-like products that must have been α' Mt appeared. Subsequent holding at high temperature produced no further changes in the microstructure. These results indicate that tempering-induced α' Mt is formed during rapid heating and not during quenching after tempering. One possible cause of tempering-induced α' Mt is the use of a salt bath, which has a much faster heating rate than a common vacuum furnace, to temper the material in a short time.

![Fig. 2](image1.png) Elevated temperature XRD profiles of 10MoA alloy.

![Fig. 3](image2.png) In-situ observation of microstructural evolution of 10MoA due to rapid lamp heating. Acicular martensite appeared within 2 s after the start of heating, indicating that martensite is formed during heating rather than cooling.
3.2 Formation conditions of tempering-induced $\alpha''_{\text{Mt}}$

The effect of the heating rate on the formation of tempering-induced $\alpha''_{\text{Mt}}$ is shown in Fig. 4. In (a), a ceramic-coated thermocouple with a wire diameter of 0.127 mm was welded to the center of a 5 $\times$ 5 $\times$ 1 mm$^3$ specimen and heated in a salt bath at 450°C. In (b), a similar specimen was wrapped ten times with aluminum foil and heated in a salt bath. In the case of (a), the temperature reached 450°C in approximately 3 s (heating rate: approximately 130°C/s), and a large amount of $\alpha''_{\text{Mt}}$ was generated by subsequent water cooling. In (b), the temperature reached 450°C in approximately 10 s (heating rate: approximately 42°C/s), and almost no $\alpha''_{\text{Mt}}$ was observed in the water-cooled microstructure. We conclude that a high heating rate is necessary to form tempering-induced $\alpha''_{\text{Mt}}$.

In a previous study, we reported that the $\alpha''_{\text{Mt}}$ formation temperature range of 10MoA (Ti-9.52Mo-6.79Al-0.052O (mass%), slightly different in composition from the present alloy) is from 300 to 550°C, and that $\alpha''_{\text{Mt}}$ does not form below 250°C; a temperature difference from room temperature (25°C) to 525°C is required for $\alpha''_{\text{Mt}}$ formation. Therefore, the effect of the temperature difference until tempering on $\alpha''_{\text{Mt}}$ formation was investigated. Two salt baths, 200°C and 550°C, were prepared. STQ specimens of approximately 5 $\times$ 5 $\times$ 1 mm$^3$ were preheated in the 200°C salt bath for 5 s, immediately tempered in the 550°C salt bath for 3 s and quenched. Figure 5 shows the microstructures before and after the two-step tempering. It can be seen that the formation of $\alpha''_{\text{Mt}}$ was suppressed in the two-step tempered sample (temperature difference of 350°C) in Fig. 5(b), compared to the tempered sample from room temperature to 550°C for 3 s (temperature difference of approximately 525°C) shown in Fig. 1(b). Also, we found that no $\alpha''_{\text{Mt}}$ was formed when the material was preheated at 300°C for 5 s and then tempered at 550°C for 3 s (temperature difference 250°C), although this is not shown in the figure.

As shown in Fig. 1, the $\alpha''_{\text{Mt}}$ transformation by tempering at 550°C for 3 s and the $\beta$-reverse transformation at 200°C for 3 min are reversible; therefore, preheating at 200°C for only 5 s should not affect the reversibility of the transformation. We conclude that the $\alpha''_{\text{Mt}}$ transformation did not occur because preheating at 200°C reduced the temperature difference to 350°C.

Tempering-induced $\alpha''_{\text{Mt}}$ formation at approximately 200°C was investigated by ensuring a temperature difference until tempering. Figure 6 shows the microstructure (a) of the STQ sample (5 $\times$ 5 $\times$ 1 mm$^3$) and the microstructure (b) of the same sample after subzero treatment with liquid nitrogen ($-196°C$ for 5 min) followed by tempering in a salt bath at 250°C for 5 s. In spite of the 446°C temperature difference in tempering, no $\alpha''_{\text{Mt}}$ was formed and even $\alpha''_{\text{Mq}}$, which had been formed in STQ, disappeared. Evidently the $\beta$ phase is metastable at approximately 200°C.

Next, to investigate the effect of specimen thickness on the formation of $\alpha''_{\text{Mt}}$, specimens with thicknesses of 2 mm and 0.1 mm were fabricated from STQ material and subjected to salt bath tempering at 450°C for 3 s. Figure 7 shows the OM microstructure before and after tempering. In the 2 mm plate sample (a), $\alpha''_{\text{Mt}}$ formed on almost the entire surface; in the 0.1 mm plate sample (b), $\alpha''_{\text{Mt}}$ did not form and even $\alpha''_{\text{Mq}}$ which was observed before tempering, disappeared. That the thickness of the specimen affects the $\alpha''_{\text{Mt}}$ transformation is
thought to be related to the thermal stress caused by the temperature difference between the surface and interior of the specimen.

3.3 Thermal stress due to heat treatment

To estimate the thermal stress generated by the salt bath tempering at 450°C, thermal conduction analysis and thermal stress analysis using the finite element method were conducted using a sample of approximately $5 \times 5 \times 1$ mm$^3$. The results are shown in Fig. 8. Using the same method as in Fig. 4, the temperature change during tempering was measured ($T_{\text{exp}}$), and the temperature change at the sample surface ($T_{\text{surf}}(t)$) was approximated using eq. (1):

$$T_{\text{surf}}(t) = T_s - (T_s - T_0) \times \exp(-at)$$  (1)

where $T_{\text{surf}}$ is the sample surface temperature, $t$ is time, $T_s$ is the salt bath temperature (450°C), $T_0$ is the initial temperature (25°C) at time $t = 0$, and $a$ is a constant (5.0). From this approximate equation, the temperature change ($T_u(t)$) inside the center of the sample ($z = 0.5$ mm) was calculated using the unsteady thermal stress finite element method. The software used was Marc 2016 from MSC Software. For the calculation, 1/8 of the sample was modeled to take symmetry into account. A hexahedral first-order element with a side of 100 µm was used as an element in the initial state. The heat conduction in the sample follows the heat conduction eq. (2):

$$\rho c \frac{dT}{dt} = \nabla \cdot (\lambda \nabla T)$$  (2)

where $\lambda$, $\rho$, and $c$ are the thermal conductivity ($\lambda = 4.85$ W/m·K), density ($\rho = 4.55$ g/cm$^3$), and specific heat ($c = 523$ J/kg·K), respectively. From the temperature gradient generated inside the sample, the thermal stresses generated in
each part were calculated assuming an isotropic linear elastic body with a coefficient of thermal expansion \( \alpha = 8.4 \times 10^{-6}/K \), Young’s modulus \( E = 50 \text{ GPa} \), and Poisson’s ratio \( v = 0.34 \). Figure 8(a) shows the measured surface temperature \( T_{\text{exp}} \), the surface temperature \( T_{\text{surf}} \) by eq. (1), the internal temperature \( T_{\text{in}} \) by eq. (2), and the temperature difference between the surface and internal \( \Delta T = T_{\text{surf}} - T_{\text{in}} \). From the figure, it was found that the inside of the specimen reached 450°C completely by tempering and holding at 450°C for 3 s. The maximum temperature difference (78.1°C) was reached at \( t = 0.1 \text{ s} \) after the sample was placed in a salt bath at 450°C, after which the temperature difference decreased.

Figure 8(b) shows the thermal stress distribution (\( \sigma_{t} \)) from the sample surface to the interior at \( t = 0.1 \text{ s} \). The compressive thermal stress at the surface of the sample is approximately \(-36 \text{ MPa} \) (approximately \(+19 \text{ MPa} \) in the interior); our hypothesis is that this thermal stress increases the \( \beta \) matrix Gibbs energy, compensates for the lack of transformation driving force, and induces the \( \alpha'_{\text{Mt}} \) transformation. For reference, with a plate thickness of 2 mm and a suitable constant \( a = 2.5 \), the maximum temperature difference (126°C) is reached at \( t = 0.28 \text{ s} \), resulting in a compressive thermal stress of approximately \(-55 \text{ MPa} \) at the sample surface (approximately \(+34 \text{ MPa} \) inside). The compressive thermal stress is high because the thermal conductivity of \( \beta \)-type Ti alloys, such as the 10MoA alloy, is very low. Therefore, if the sample is heated at a low speed, or if the temperature difference up to the tempering temperature is small, or if the sample is thin, the \( \alpha'_{\text{Mt}} \) transformation does not occur because large thermal stresses do not occur inside and outside the sample.

Figure 9 shows the results of high-temperature tensile tests carried out to investigate whether this thermal stress is high enough to generate a work-induced \( \alpha'_{\text{Md}} \). In general, the higher the tensile temperature, the higher the stress value.
required for work-induced transformation, and if this value exceeds the critical stress value for slip deformation, work-induced martensite will not form. In our tensile tests at room temperature, the work-induced transformation stress (yield stress in the first stage) was approximately 250 MPa; it was approximately 400 MPa in the tensile tests at 150°C. Under tension at 200°C, yielding was observed at approximately 550 MPa, which was attributed to slip deformation. These results suggest that thermal stress may promote the a’M transformation but is too small to induce it alone. A tensile test at 450°C was not performed, because a’iso forms in a very short time at 450°C, and the material hardens significantly.

3.4 Distribution of a’M in a sample

Rapid heating to 450°C resulted in the formation of a’M over the entire surface of the specimen, while rapid cooling from 1050°C resulted in the formation of only a small amount of a’M. To investigate this phenomenon, the same thermal stress analysis used in Section 3.3 was applied to the STQ treatment. When a plate specimen with a thickness of 1 mm was quenched from 1050°C into ice water, the maximum temperature difference ΔT = −283°C occurred at t = 0.08 s. This resulted in a tensile thermal stress of approximately +130 MPa on the specimen surface and a compressive thermal stress of approximately −66 MPa inside the specimen. These results indicate that only a small amount of a’Mq was produced in the STQ material, even though STQ generates more than twice the thermal stress of 450°C tempering.

Cross-sectional observations were carried out on the STQ and the sample tempered at 450°C for 3 s to investigate the distribution of a’M. Figure 10 shows the cross-sectional microstructure of the STQ sample (approximately 5 × 5 × 1 mm³). Almost no a’Mq was observed near the surface of the sample, but a large amount of a’Mq was observed inside it. On the other hand, the cross-sectional microstructure of the tempered sample (thickness: 2 mm) obtained from the sample used in Fig. 7(a) is shown in Fig. 11; in this sample, a’M was produced primarily near the surface with less inside. The reversal of the a’M distribution between the STQ and tempered samples can be attributed to the effect of thermal stress: in both heat treatments, a’M occurred on the side where compressive thermal stress occurred. Using the lattice constants of the β-phase and a’M on the sample tempered at 550°C for 3 s as described in Section 3.1, the volume change associated with the a’M transformation is calculated to be $V_{a’M} - V_{β}$ = −0.3% volume shrinkage. Since the a’M transformation decreases the sample volume, the transformation should be favored by compressive thermal stress. Recently, Qi et al. also reported that the microstructure of Ti–10V–2Fe–3Al STQ material consists mostly of β phase, with only a small amount of a’Mq on the sample surface, while the sample interior consists of a large amount of a’Mq and a small amount of β phase. They believe that the formation of surface a’Mq is suppressed because interstitial atoms (such as oxygen) in the surface layer are enriched during solution treatment. A similar surface enrichment effect may have occurred in the 10MoA STQ samples used in this study, but this possibility seems unlikely because abundant a’M is induced in the surface layer by the subsequent rapid tempering.

4. Conclusion

Investigation of the formation mechanism of a’M produced by STQ and rapid short-time tempering of 10MoA alloy clarified the following points:

1. The surface microstructure of STQ material is composed of β phase and a small amount of a’Mq, and a’M is formed by tempering from 450°C to 550°C.
for 3 s. However, $\alpha''_{\text{Mf}}$ is reversibly transformed to $\beta$ phase by aging at 200°C. 

(2) **In-situ** microstructure observation by rapid ramp heating showed that the tempering-induced $\alpha''_{\text{Mf}}$ was formed during heating rather than quenching.

(3) When tempering at 450°C, $\alpha''_{\text{Mf}}$ is not formed under any of the following conditions: slow heating rate (42°C/s); small temperature difference from the initial up to the tempering temperature (less than 250°C); thin specimen thickness (0.1 mm).

(4) When a plate sample with a thickness of 1 mm is tempered in a salt bath at 450°C, a compressive thermal stress of about $-36 \text{ MPa}$ is generated on the sample surface, which promotes the $\alpha''_{\text{Mf}}$ transformation with volume shrinkage.

(5) Even if the conditions of heating rate, temperature difference, and specimen thickness are met, tempering at around 200°C will never form $\alpha''_{\text{Mf}}$, but will result in $\beta$ single phase.

(6) In STQ, $\alpha''_{\text{Mf}}$ is formed inside the specimen, while $\alpha''_{\text{Mf}}$ due to tempering is formed in the specimen surface layer. We infer that this result is related to the compressive thermal stress generated during heat treatment.

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