Kinetic Study on the Crystal Transformation of Fe-Doped TiO$_2$ via In Situ High-Temperature X-ray Diffraction and Transmission Electron Microscopy

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INTRODUCTION

Titanium dioxide (TiO$_2$) is a very widely used material that cannot easily undergo chemical changes. Currently, its two crystal types, which are used in many industrial applications, are anatase (A) and rutile (R). Anatase can undergo an irreversible phase change to rutile under certain conditions. Rutile exhibits a better crystal structure, denser atomic arrangement, higher dielectric constant, stronger ultraviolet (UV) shielding ability, and better stability than anatase. Because the structure of rutile is denser than that of anatase, it exhibits little distortion; therefore, its catalytic ability is poorer than that of anatase. Different crystal types show different properties, functions, and applications. In the production process of TiO$_2$, generally, raw ore is directly used; hence, Fe impurities cannot be avoided, which somewhat affect the crystal transformation of TiO$_2$. The phase transition process of TiO$_2$ continuously changes with time and temperature. Under normal pressure, only anatase $\rightarrow$ rutile phase transition occurs; the phase transition temperature is approximately 600 °C. Higher temperature or longer holding time leads to a more complete transformation from anatase to rutile. Because rutile is the most stable phase, this phase transition process is irreversible. The application prospects of TiO$_2$ depend on its crystal type, and a reasonable process is required to prepare TiO$_2$ using a suitable crystal type to achieve its optimal performance in application areas. Therefore, understanding the stability of the TiO$_2$ crystal form and its crystal transformation kinetics and determining techniques to control the transformation process to achieve single-phase or multiphase TiO$_2$ are very important.

In this study, Fe-doped TiO$_2$ was synthesized via the hydrolysis and coprecipitation of titanium oxide sulfate (TiOSO$_4$) and ferric chloride (FeCl$_3$$\cdot$6H$_2$O). Additionally, TiO$_2$ with varying Fe contents was calcined using in situ high-temperature X-ray diffraction (XRD) technology to simulate the real-time reaction process of the actual titanium mineral-calcining reaction system. Moreover, Rietveld full-spectrum fitting, Williamson–Hall, and other methods were used to study the kinetics of the Fe to TiO$_2$ crystal transformation process under high-temperature conditions. Research results showed that the addition of Fe to TiO$_2$ increases the transformation temperature of TiO$_2$ anatase to rutile and accelerates the anatase $\rightarrow$ rutile transformation process. Further, Fe-doped TiO$_2$ agreed well with the Johnson–Mehl–Avrami–Kohnogorov (JMAK) phase transition kinetic model. The JMAK formula showed that the TiO$_2$ transformation kinetic model matched the Johnson–Mehl–Avrami–Kohnogorov (JMAK) model and that its phase transition was affected by crystal defects. Finally, Fe$^{3+}$ in Fe-doped TiO$_2$ was reduced to Fe$^{2+}$ to generate oxygen vacancies, thus promoting the rate of transformation from titanium ore to rutile.
phase transition process was affected by crystal defects. The addition of Fe was conducive to the generation of vacancy defects, and the defects accelerated the transformation speed, thus accelerating the anatase $\rightarrow$ rutile transformation process.25

**EXPERIMENTAL SECTION**

TiOSO$_4$ and FeCl$_3$·6H$_2$O were hydrolyzed and precipitated to prepare the Fe-containing TiO$_2$ precursor. The samples were doped with Fe impurities with doping amounts of $x = 0, 0.5, 1, 2.5, 5$, and 7.5, termed TF0, TF1, TF2, TF4, TF7, and TF10, respectively. First, titanium tetrachloride (TiCl$_4$) was subjected to an ice-water bath and then added to a sulfuric acid solution. Then, the mixture was stirred until it was completely dissolved, thereby forming a TiOSO$_4$ solution. Thereafter, a certain amount of FeCl$_3$·6H$_2$O was added to the TiOSO$_4$ solution by the mass ratio, and the mixture was stirred. The pH of the solution was adjusted to 6 using a saturated NaOH solution to form solid precipitates. The formed precipitate was washed several times with distilled water until SO$_4^{2-}$ and Cl$^-$ could not be detected when saturated BaCl$_2$ and AgNO$_3$ solutions were used. Then, to obtain the Fe-containing TiO$_2$ precursor, the washed precipitate was dried in an electrically heated blast

![Figure 1](http://pubs.acs.org/journal/acsodf)

Figure 1. In situ high-temperature XRD patterns of (a) TF0, (b) TF1, (c) TF2, (d) TF4, (e) TF7, and (f) TF10.
RESULTS AND DISCUSSION

In Situ High-Temperature XRD Phase Transition Analysis. Figure 1a–f shows the in situ high-temperature XRD patterns of the six samples (TF0–TF10). The test started at 300 °C, and it was performed at an interval of every 25 °C until reaching 1000 °C. However, during the cooling process, the test was performed at an interval of 200 °C. The diffraction peak appearing at 2θ = 45° (Figure 1a–f) was formed after the platinum bar of the heated sample was exposed, and it does not correspond to the TiO2 diffraction peak. In the subsequent analysis, this nonsample peak was eliminated. At 300 °C, TiO2 was still in its anatase phase. With an increase in temperature, the diffraction peak intensity of the anatase phase gradually increased. In the vicinity of 2θ = 37, 55, and 72°, multiple anatase phase peaks were observed with relatively large peak widths. The rutile phase began to appear after 600 °C. When the temperature was further increased, the XRD peak of the anatase phase became weak until it disappeared. Moreover, the diffraction peak of the rutile phase continued to become strong, and the peak width became narrow, indicating that the rutile phase was continuously growing. Compared with the non-Fe-doped samples, after increasing the temperature to a certain phase was continuously growing. Compared with the non-Fe-doped samples, the peak width became narrow, indicating that the rutile phase was determined using the Rietveld full-spectrum fitting method and the Williamson–Hall mapping method. The crystal structure of the sample was observed using a high-resolution transmission electron microscope (Tecnai G2 F20).

Table 1. Phase Transition Temperature of TiO2 with Different Fe Contents

|        | TF0 | TF1 | TF2 | TF4 | TF7 | TF10 |
|--------|-----|-----|-----|-----|-----|------|
| R appear. | 600 | 625 | 675 | 675 | 675 | 675  |
| A disappear. | 825 | 800 | 825 | 825 | 825 | 825  |
| FTO appear. | 800–cooing | 900 | 775 | 725 | 725 | 725  |

It can be seen from Table 1 that the R phase transition temperature of TF0 was 600 °C and that the Fe addition increases the appearance temperature of the R phase. When the doping amount was ≥1.15% (TF2), the rutile phase transition temperature was maintained at 675 °C. The temperature at which the anatase phase disappeared did not change considerably. Next, all samples were maintained between 800 and 825 °C; however, because of the Fe doping, the temperature range of the anatase → rutile transition became smaller, 225 and 175 °C for TF0 and TF1, respectively, and 150 °C for TF2, TF4, TF7, and TF10.

Figure 2a–d shows the XRD patterns before and after the formation and disappearance temperatures of the TF0 phase. The XRD patterns of TF0 revealed only the anatase phase at 575 °C. When the temperature was increased to 600 °C, a rutile phase diffraction peak was observed at 2θ = 27.4°, which was the strongest peak of the rutile phase and first appeared during rutile phase transition. Thus, the temperature at which this peak began to appear in the rutile phase is the TiO2 phase transition start temperature. Similarly, the temperature at which all diffraction peaks of the anatase phase disappeared was the termination temperature of the TiO2 phase transition. Generally, the characteristic peak of the anatase phase, which disappeared last, was observed at 2θ = 25.3°, which is the strongest peak of the anatase phase (Figure 2b). When the temperature reached 825 °C, only the diffraction peak of the rutile phase remained. The change temperature of each phase in TF0–TF10 was obtained, as shown in Table 1.
For the Fe-doped samples, as the Fe content increased, the formation temperature of the FTO phase decreased, indicating that the formation of Fe₂TiO₅ is rapid when the Fe content is increased. Moreover, the rutile phase of TF1 started to rapidly increase after the temperature was increased by 50 °C and that of the rest of the samples started from 675 °C. At the beginning of the anatase $\rightarrow$ rutile phase transition, at 700 °C, the samples entered a rapid growth stage; then, they steadily increased after 750 °C until the anatase $\rightarrow$ rutile phase transition was complete. After reaching 850 °C, the relative content of the rutile phase slightly decreased.

Table 2 shows the relative content of each phase in the phase transition process of Fe-doped TiO₂. By analyzing the relative change in the content, it can be inferred that Fe₂TiO₅ was formed by the reaction of rutile TiO₂ and Fe and that the addition of Fe increased the temperature of the anatase $\rightarrow$ rutile phase transition but reduced the temperature range of the transition process. The end temperature of the sample anatase $\rightarrow$ rutile transition basically ended at the same temperature of 825 °C.

For further investigation, to study the holding time effect on the rutile TiO₂ content, the samples with the different Fe

![Figure 3](image-url)
contents were tested using high-temperature in situ XRD. The samples were retained at their starting phase transition temperatures (Table 1), and a test was performed once every 10 min from the beginning to the temperature. Each test time was 10 min, for a total of 5 h. Figure 4 presents the obtained in situ XRD pattern of the holding time.

Figure 4. In-situ XRD diagram of each sample at phase change temperature.
It can be seen from Figure 4 that at the phase transition temperature, the sample with the low iron content demonstrated a little phase variation during the same holding time. TF0 (600 °C) exhibited only 6.3% of the rutile phase at the end of the holding time.

Figure 5. Content of the rutile phase of (a) TF0, (b) TF1, (c) TF2, (d) TF4, (e) TF7, and (f) TF10 at the phase transition temperature, 700 °C, and 750 °C.

Figure 6. Different Fe content-doped TiO2 kept warm at phase change temperature and the relative content of each phase changing over time: (a) anatase-A and pseudobrookite-FTO and (b) rutile-R.
holding time, while TF1 (625 °C) exhibited 8%. The samples with high Fe contents exhibited more of the rutile phase, and the rutile phase content in TF10 (675 °C) reached 72.6% at the end of heat preservation. Although the temperature at which the Fe-doped samples started undergoing the anatase → rutile phase transition was high, the heat preservation results further indicated that Fe doping increased the reaction rate. When retained at 750 °C, all samples achieved reaction equilibrium after 50 min. The rutile phase content was calculated using the Rietveld full-spectrum fitting and Williamson–Hall mapping methods, and the calculation results are shown in Figure 5a–f.

**Kinetic Analysis of the Crystal Transformation of Fe-Doped TiO₂.** The holding phase content of different TiO₂ samples was analyzed with respect to time at different temperatures, and the kinetic parameters of the TiO₂ phase transition were solved using the change in the rutile phase. First, the change in TiO₂ was analyzed while holding the phase transition temperature to determine the phase transition kinetic model of Fe-doped TiO₂. Figure 6 shows the corresponding change in the phase content.

As shown in Figure 6a, within 5 h, the TF10 sample formed an FTO phase after 80 min; the other Fe-doped samples did not form FTO phases. It can be seen from Figure 6a that in the early stage of holding TF10, the FTO phase began to appear from 75 min because the formation of the FTO phase can lead to excessive defects in the crystal. Moreover, the rutile phase growth decreased, indicating the presence of excessive Fe; hence, TF10 could no longer drive the anatase → rutile phase transition at this temperature. In Figure 6a, the anatase phase of TF10 decreased with time. Furthermore, TF7 tended to decelerate; however, the Fe amount was relatively small, and the FTO phase transition did not occur yet. In Figure 6b, the different samples were retained at the temperature at which their phase transition began. Moreover, the rutile phase increased with the Fe content and in the slope of the rutile phase growth. The phase content of TF0–TF4 linearly increased within 5 h of the holding time. After TF7 and TF10 rapidly grew at the beginning, their growth began to decelerate, the relative content growth curve of the rutile phase became slower and flatter, and the TF10 performance became more obvious.

The matching of the phase transition kinetics of TiO₂ and the solution of the related parameters generally use the standard first-order kinetic model

\[(\ln(1 - \alpha) = -kt)\]

or the JMAK model

\[(\ln[-\ln(1 - \alpha)] = n \ln t + \ln k)\]

In the above equation, \(t\) is the time, \(\alpha\) is the mass fraction of the phase transition, \(k\) is the kinetic reaction rate constant, and \(n\) is the Avrami index, which is related to the phase transition mechanism.

![Figure 7. Kinetic model fitting: (a) standard first-order kinetic model and (b) JMAK model.](https://dx.doi.org/10.1021/acsomega.0c05609)
The above two models were used to fit the insulated samples, and the kinetic model fitting diagram was obtained, as shown in Figure 7. By comparing the goodness of fitting rutile in Figure 7a and b, TF0, TF1, TF2, TF4, and TF7 better fit the standard first-order kinetic model, TF10 better matches the JMAK model than the standard first-order kinetic model, and TF4 and TF7 show good matching degrees with the two models. Overall, the matching of the two models was relatively good. Generally, a low Fe content or pure TiO2 conforms to the first-order kinetic model of nucleation growth. When the impurity concentration was high, other growth methods were observed. The Fe content in TF4, TF7, and TF10 was relatively high. Some problems occurred when these samples were fitted with the JMAK model. Next, the Avrami index $n$ of the JMAK formula is related to the phase transition reaction mechanism, and $n$ is the slope of the equation obtained by fitting. When $n = 1$, the JMAK model corresponds to a first-order kinetic model.26 However, when $n = 2/3$, the phase transition is related to crystal defects.

Figure 8. $\ln(-\ln(1 - \alpha))$-$\ln$ diagram of holding time at various temperatures (a) TF0, (b) TF1, (c) TF2, (d) TF4, (e) TF7, and (f) TF10.
Based on the $\ln[-\ln(1-\alpha)]-\ln t$ curve diagram obtained using the JMAK kinetic equation, the kinetic reaction rate constant $k$ can be calculated. From the Arrhenius equation, the reaction rate and temperature show the following relationship

$$k = k_0 \exp\left(-\frac{E}{RT}\right)$$

In the formula, $k_0$ is a constant, $R$ is the general gas constant, and $T$ is the isothermal absolute temperature. By considering the natural logarithm of both sides of the above formula, we achieve

$$\ln k = \ln k_0 - \frac{E}{RT}$$

After plotting an $\ln k-1/T$ curve, the linear regression fitting can yield the activation energy of the TiO$_2$ phase transition.

It can be seen from Figure 5 that with the increase in temperature, the time required for TiO$_2$ to reach the A $\rightarrow$ R phase transition equilibrium became shorter. Further, a high Fe content leads to a short transition equilibrium time. The change rule was similar to the JMAK model; hence, $\ln[-\ln(1-\alpha)]-\ln t$ was used for plotting as shown in Figure 8a$-f$, and the obtained $\ln k$ value and phase change activation energy are shown in Table 3.

| samples | temperature (K) | $\ln k$ | $E$ (kJ/mol) |
|---------|----------------|---------|--------------|
| TF0     | 873            | -5.93375| 212.30       |
|         | 973            | -4.97477|              |
|         | 1023           | -1.01169|              |
| TF1     | 898            | -6.3588 | 28.32        |
|         | 973            | -9.67976|              |
|         | 1023           | -5.2047 |              |
| TF2     | 948            | -9.81289| 741.63       |
|         | 973            | -14.6201|              |
|         | 1023           | -4.22434|              |
| TF4     | 948            | -10.3711| 759.97       |
|         | 973            | -10.8263|              |
|         | 1023           | -3.83374|              |
| TF7     | 948            | -9.75908| 550.51       |
|         | 973            | -3.1779 |              |
|         | 1023           | -3.77046|              |
| TF10    | 948            | -5.92342| 451.57       |
|         | 973            | -6.90546|              |
|         | 1023           | -2.16794|              |

The findings of the kinetic study revealed that the phase transition process of Fe-doped TiO$_2$ was mainly controlled by crystal defects. Because the ionic radius of Fe$^{3+}$ (0.064 nm) is smaller than that of Ti$^{4+}$ (0.061 nm), Fe$^{3+}$ can enter TiO$_2$ instead of Ti$^{4+}$ to form a solid solution. Moreover, because Fe is a variable element, Fe$^{3+}$ can be reduced to Fe$^{2+}$ during the phase change; thus, the produced Fe$^{2+}$ can form more oxygen vacancies. Vacancy defects promote the conversion of anatase to rutile, which was mainly reflected in the conversion rate of the A $\rightarrow$ R phase transition. In metal-cation-doped TiO$_2$, the cation generally enters the TiO$_2$ lattice to replace Ti$^{4+}$ to form a replacement solid solution. When the ion radius significantly differs from the Ti$^{4+}$ radius, the TiO$_2$ lattice will be distorted, and excess energy will be stored. Anatase will first release this part of energy during the phase transition and then transform into rutile. After the Fe doping, the A $\rightarrow$ R transition temperature increased. After analysis, the samples with low Fe contents well fitted the standard first-order kinetic model, the samples with high Fe contents well matched the JMAK model, and the phase transition process of the Fe-doped samples was controlled by the crystal defects. The activation energy of the Fe-doped samples was higher than that of the non-Fe-doped TiO$_2$ sample.

Figure 9a$-d$ shows the TEM image of TF0 and TF4, which were calcined at 700 °C. As shown in the figure, the phase observed using a transmission electron microscope at 700 °C mainly corresponded to the anatase phase. From the XRD analysis results, this phase was observed at 700 °C. The rutile phase already appeared underneath; however, the rutile amount was relatively small at 700 °C; therefore, observing the rutile phase lattice fringes under the TEM is difficult. It can be seen from Figure 9a$-d$ that the average grain size of the Fe-doped sample TF4 is smaller than that of TF0.

Figure 10a,b shows the TEM images of TF0 and TF4 when calcined at 800 °C. In the figure, the interplanar spacing $d = 0.246$ nm was close to the spacing of the rutile phase (101) crystal plane. Moreover, $d = 0.32$ nm was close to the rutile phase (110) spacing. At 800 °C, the anatase phases of TF0 and TF4 were basically transformed into rutile phases.

From the above findings, it can be inferred that Fe-doped TiO$_2$ was transformed from the anatase phase to the rutile phase. Next, the prepared precursor TiO$_2$ mainly comprised anatase crystals with a relatively small number of crystal grains, in addition to non-Fe-doped and Fe-doped crystals. The grain size
The addition of Fe increased the TiO2 anatase amount further decreased the size of the crystal particle. The initial grain size was below 10 nm. Increasing the Fe doping made the phase transition more obvious. However, Fe accelerated the A → R phase transition process. Before the A → R transformation, Fe was in the form of Fe3+ in TiO2. After the phase transition began, Fe3+ was reduced to Fe2+, forming more vacancy defects and accelerating the A → R phase transition process. After analyzing the phase transition kinetics, we found that Fe-doped TiO2 well matched the JMAK model. The index n ≈ 0.6 in the JMAK model showed that the phase transition process of TiO2 was affected by defects. Finally, the addition of Fe was conducive to the generation of vacancy defects, which accelerated the rate of the phase change.

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Notes
The authors declare no competing financial interest.

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■ REFERENCES

(1) Beltrán, A.; Gracia, L.; Andris, J. Density functional theory study of the brookite surfaces and phase transitions between natural titania polymorphs. J. Phys. Chem. B 2006, 110, 23417−23423.

(2) Ghosh, T. B.; Dhabal, S.; Datta, A. K. On crystallite size dependence of phase stability of nanocrystalline TiO2. J. Appl. Phys. 2003, 94, 4577−4582.

(3) Borisov, A.; Aranovich, L. Rutile solubility and TiO2 activity in silicate melts: An experimental study. Chem. Geol. 2020, 119817.

(4) Ranjith, R.; Renganathan, V.; Chen, S. M.; Selvan, N. S.; Rajam, P. S. Green synthesis of reduced graphene oxide supported TiO2/Co3O4 nanocomposite for photocatalytic degradation of methylene blue and crystal violet. Ceram. Int. 2019, 12926.

(5) Medvids, A.; Varnagiris, S.; Letko, E.; Milcius, D.; Grase, L.; Gaidukovs, S.; Mychko, A.; Pludons, A.; Onufrijevs, P.; Mimura, H. Phase transformation from rutile to anatase with oxygen ion dose in the TiO2 layer formed on a Ti substrate. Mater. Sci. Semicond. Process. 2020, 106, 104776.

(6) Mohamad, M.; Ul Haq, B.; Ahmed, R.; Shaari, A.; Ali, N.; Hussain, R. A density functional study of structural, electronic and optical properties of titanium dioxide: Characterization of rutile, anatase and brookite polymorphs. Mater. Sci. Semicond. Process. 2015, 31, 405−414.

Figure 10. TEM images of (a) TF0 and (b) TF4 after calcination at 800 °C.
Enhanced visible light photocatalytic properties of Fe-doped TiO2 nanorod clusters and monodispersed nanoparticles. Appl. Surf. Sci. 2011, 257, 8121−8126.

Anitha, B.; Khadar, M. A. Anatase-rutile phase transformation and photocatalysis in peroxide gel route prepared TiO2 nanocrystals: Role of defect states. Solid State Sci. 2020, 106392.

Wang, H.; Zhang, N.; Cheng, G.; Guo, H.; Shen, Z.; Yang, L.; Zhao, Y.; Alsaedi, A.; Hayat, T.; Wang, X. Preparing a photocatalytic Fe-doped TiO2/rGO for enhanced bisphenol A and its analogues degradation in water sample. Appl. Surf. Sci. 2020, 505, 144640.

Süleyici Cergel, M.; Demir, E.; Atay, F. The effect of the structural, optical, and surface properties of anatase-TiO2 film on photocatalytic degradation of methylene blue organic contaminant. Ionics 2019, 25, 4481−4492.

Ismail, M. A.; Memon, N. K.; Hedhili, M. N.; Anjum, D. H.; Chung, S. H. Synthesis of TiO2 nanoparticles containing Fe, Si, and V using multiple diffusion flames and catalytic oxidation capability of carbon-coated nanoparticles. J. Nanopart. Res. 2016, 18, 1−14.

Hanaor, D. A. H.; Sorrell, C. C. Review of the anatase to rutile phase transformation. J. Mater. Sci. 2011, 46, 855−874.

Shui, M.; Song, Y.; Wang, Q.; Ren, Y. Thermal behavior, microstructure, phase transformation, and crystal growth kinetics of nano-scale Fe3+-doped TiO2 xerogel powders. Curr. Appl. Phys. 2010, 10, 1360−1365.

Huang, X.; Tian, C.; Qin, H.; Guo, W.; Gao, P.; Xiao, H. Preparation and characterization of Al3+-doped TiO2 tight ultrafiltration membrane for efficient dye removal. Ceram. Int. 2020, 46, 4679−4689.

Singh, V.; Rao, A.; Tiwari, A.; Yashwanth, P.; Lal, M.; Dubey, U.; Aich, S.; Roy, B. Study on the effects of Cl and F doping in TiO2 powder synthesized by a sol-gel route for biomedical applications. J. Phys. Chem. Solids 2019, 134, 262−272.

Li, X.; Chen, Z.; Shi, Y.; Liu, Y. Preparation of Fe co-doped TiO2 with visible light response. Powder Technol. 2011, 207, 165−169.

Kolenko, Y. V.; Burukhin, A. A.; Churagulov, B. R.; Oleynikov, N. N. Synthesis of nanocrystalline TiO2 powders from aqueous TiOSO4 solutions under hydrothermal conditions. Mater. Lett. 2003, 57, 1124−1129.

Kaschel, F. R.; Vijayaraghavan, R. K.; Shmeliov, A.; McCarthy, E. K.; Canavan, M.; McNally, P. J.; Dowling, D. P.; Nicolosi, V.; Celikin, M. Mechanism of stress relaxation and phase transformation in additively manufactured Ti-6Al-4V via in situ high temperature XRD and TEM analyses. Acta Mater. 2020, 188, 720−732.

Londono-Restrepo, S. M.; Herrera-Lara, M.; Bernal-Alvarez, L. R.; Rivera-Muñoz, E. M.; Rodriguez-Garcia, M. E. In-situ XRD study of the crystal size transition of hydroxyapatite from swine bone. Ceram. Int. 2020, 46, 24454−24461.

Goerens, J.; Manninger, T.; Goetz-Neunhoeffer, F. In-situ XRD study of the temperature-dependent early hydration of calcium aluminate cement in a mix with calcite. Cem. Concr. Res. 2020, 136, 106160.

Zhang, H.; Jerusalem, A.; Salvati, E.; Papadaki, C.; Fong, K. S.; Song, X.; Korsunsky, A. M. Multi-scale mechanisms of twinning-dewinning in magnesium alloy AZ31B simulated by crystal plasticity modeling and validated via in situ synchrotron XRD and in situ SEM-EBSD. Int. J. Plast. 2019, 119, 43−56.

Okada, K.; Yamamoto, N.; Kameshima, Y.; Yasumori, A.; MacKenzie, K. J. D. Effect of Silica Additive on the Anatase→Rutile Phase Transformation. J. Am. Ceram. Soc. 2001, 84, 1591−1596.

Topuz, B. B.; Gündüz, G.; Mavis, B.; Colak, U. The effect of tin dioxide (SnO2) on the anatase-rutile phase transformation of titania (TiO2) in mica-titania pigments and their use in paint. Dyes Pigments. 2011, 90, 123−128.

Wanta, K.; Loakit, L.; Khemthong, P.; Grisdanurak, N.; Fukaya, K. Calculation temperature effect on solvothermal Fe-TiO2, and its performance under visible light irradiation. J. Taiwan Inst. Chem. Eng. 2010, 41, 612−616.

Wang, C. L.; Hwang, W. S.; Chu, H. L.; Lin, H. J.; Ko, H. H.; Wang, M. C. Kinetics of anatase transition to rutile TiO2 from titanium dioxide precursor powders synthesized by a sol-gel process. Ceram. Int. 2016, 13136−13143.