Phase transformation and microstructure evolution study in various alloy systems: An insight

S M. Jagadeesh Babu, Pramod S. Kataraki,*, K S. Narayanaswamy

School of Mechanical Engineering, Reva University, Rukmini Knowledge Park, Kattigenahalli, Yelahanka, Bengaluru – 560064, Karnataka state, India

*Corresponding author: Pramodkumar.sk@reva.edu.in

Abstract. Phase transformations occurring in various metals like steel, titanium and super alloys are integrated and presented in this paper. In steels, the microalloying with Nb and Nb-Mo decrease in transformation temperature indicates the acceleration of transformation kinetics which affect the microstructure and continuous cooling transformation (CCT) diagram. Incase of titanium alloys, the thermohydrogen treatment (THT) uses hydrogen as temporary alloying element which facilitates the alloys to be processed at lower stresses and/or temperatures. Thermomechanical processing schedules are designed based on the β transus temperature to produce a variety of novel microstructures. In the case of crystalline materials, plastic deformations assisted by dislocations and the concentration of dislocation increases with increasing plastic strain thus becoming a precursor of twinning. Stress-strain curves for different alloys suggested the microstructural changes account for deformation induced twinning and martensite transformation.

1. Introduction

Metals comprise physical properties like electrical conductivity, magnetic susceptibility etc. and also exhibit different behaviours. Metals occupy maximum area in the periodic table and pure metals crystallize into various structures (hcp, fcc, bcc etc.) [1]. Phase transformation (solid-state) is a phenomenon during which the crystals change their states (initial to final). Phase transformations occur when the material undergoes heat treatment or warm work or thermomechanically processed in single or two phase region. Usually, high temperature crystal structure differs from that of room temperature for e.g., in titanium or zirconium the high-temperature BCC phase transforms to room temperature HCP structure [2]. Basically, phase transformations are understood by analyzing continuous cooling transformation (CCT) diagram. The time–temperature–transformation/precipitation (TTT/TTP) diagrams combined with the continuous-cooling-transformation/precipitation (CCT/CCP) diagrams are used over the years to represent the kinetics of phase transformations especially in steels [3]. The solid-state phase transformation modifies microstructure and improves both physical and mechanical properties. The driving force of the solid-state phase transformations originate from the atomic level electronic transfer between d-electrons and s-band [4]. Studies have analysed phase transformation and microstructure using dilatometry tests and electron backscattered diffraction (EBSD). The near β or α+β titanium alloys belong to the category of heat treatable alloys, so novel microstructures are obtained through thermomechanical process (warm or hot working) which results in improvement in mechanical properties. Microstructure modification through addition of micro alloying results in increase in strength, solid solution
strengthening and precipitation hardening [5]. Phase transformation and the microstructure depend on factors such as chemical composition of the material, deformation, heat treatment time and temperature [6]. β titanium alloys exhibit superior properties in terms of its mechanical behavior attribute to dislocations slip and twinning, deformation induced phase transformations (α, α′′ phases) mechanisms [7]. In view of the above, an insight was made to unite the various aspects of phase transformations in different alloy systems.

2. Phase transformation through microstructure refinement

Isasti et al.[5] studied microstructural characterization and phase transformation of low-carbon steels using electron backscattered diffraction (EBSD) and dilatometry tests. The effects of alloying additions and deformation on the CCT diagram and microstructure was analyzed. The obtained microstructures comprised ferrite, pearlite, bainite and martensite depending on cooling rates. Microstructures of 6NbMo31 steel comprising polygonal ferrite (PF) + degenerated pearlite (DP) for cooling rate 0.1 K/s (Fig. 1(a, c)) and bainitic ferrite + martensite for the cooling rate 100 K/s (Fig. 1(b, d)) are shown in Fig. 1.

Figure 1. Microstructures of 6NbMo31 steel for the cooling rates (a) 0.1 K/s and (b) 100 K/s from Scanning Electron Microscopy and corresponding (c,d) Electron Back Scatter Diffraction (EBSD) inverse pole figures of (a,b) respectively [5] (e) Illustration of TTT diagram for Titanium based alloys [6].

The alloying additions of Mo and Nb promoted a shift in the CCT diagrams to lower transformation temperatures indicating the acceleration of transformation kinetics. The complex interactions between the phases formed, chemical composition and deformation has an effect on microstructure and is obvious from Fig. 1(e). From classical phase transformation theory, the ‘n’ values represent the various dominating mechanisms depending on the working temperature range. Bein and J. Beche[6] studied the occurrence of isothermal phase transformations in Ti-based alloys so as to distinguish the kinetics and the process of precipitation. It was suggested that the attempt to determine the phase transformations through electrical resistivity measurement approach found to be efficient and lead to develop TTT diagram for the titanium alloys[6]. A special heat treatment technology Thermohydrogen treatment (THT) uses hydrogen as a temporary alloying element which modifies phases and kinetics of phase transformations in Ti-based alloys. THT process allows the titanium alloy to: (a) processed at lower stresses and/or lower temperatures, and (b) heat-treated to produce novel microstructures with enhanced mechanical properties. Generally, the microstructure refinement in titanium alloys after THT is caused by hydrogen induced phase transformation viz., transformation of α/β, formation of hydride δ and martensites α′ and/or α″ [8].
In case of two phase (α+β) titanium alloys, the microstructures are characterized by the grains (α phase network) of the prior β phase on the boundaries (Fig. 2a). The colonies of α lamellae inside the grains nucleate mainly on the grain boundaries during the phase transformation process [9].

![Figure 2](image-url) (a) Illustration of two-phase (α+β) microstructure in titanium alloys [9]
(b) Microstructure evolution during β → α phase transformation in Ti-6Al-4V+Boron (B) alloy [2].

Figure 2b shows the modified mechanism of β → α phase transformation due to the addition of boron in Ti-6Al-4V alloy [2]. Usually, titanium alloys are deformed at temperatures corresponding to (α + β) or β-phase regions and subsequent heat treatment is carried out below or above β-transus (temperature at which α + β → β phase transformation occurs) corresponding to the occurrence of α → β → α phase transformation. The low-temperature behavior of titanium alloys depend on the β phase microstructure at high-temperature and its phase transformation [2]. Thermomechanical processing parameters are designed based on the β transus temperature. The variety of novel microstructure evolution with variety of morphology depends upon prior processing history with respect to β transus temperature (above or below). Primary processing (above the β transus) involves the breakup of cast microstructure leading to the formation of lamellar colonies of α and β similar to Fig. 2a. Whereas, secondary processing (below the β transus) involves the evolution of lamellar to equiaxed microstructure. The lamellar microstructure known to exhibit high strength and fracture toughness, the equiaxed microstructure possesses good ductility and low-cycle fatigue property [10].

Xie et al. [11] investigated phase transformation by thermo-dilatometry as shown in Fig. 3. The presence of higher alloying content in the alloy and the intercritical annealing decreased the Ac1 temperature thereby stabilized the austenite to as low as 680 °C.

![Figure 3](image-url) (a) Illustrating the effect of intercritical annealing on Ac1 temperature through dilatometer
curves. (b) Transmission Electron Microscope (TEM) micrographs to show the retention of austenite between ferrite and martensite. (c) Scanning Electron Microscope (SEM) micrograph showing microvoids (white arrows) at the martensite and ferrite interface [11].

TEM micrographs show fine retained austenite between body-centre-cubic (BCC) ferrite matrix laths and the selected area diffraction (SAD) results confirmed the coherent orientation relationship of austenite with that of annealed martensite ferrite, Fig. 3b[11]. In addition, the fractographs after Charpy test observed by SEM are shown in Fig. 3c, which suggested that the greater stress and dislocations get accumulate at the interface of the ferrite and martensite, also lead to the nucleation of predominant microvoids, Fig. 3c [11].

Figure 4. (a) Microstructure evolution as a function of strain during the deformation of 304 stainless steel (b) Schematic illustration of the deformation process and the respective changes in the phases for the 304 stainless steel [12].

The true stress-true strain curve separated into different regions based on microstructural changes for a 304 stainless steel is shown in Fig 4. TEM images (Fig 4a) revealed that the increase in the amount of twins and martensite with strain indicates phase transformation taking place during straining. At the strain value of 57%, the martensite fraction is ∼53% and that of twin is 45% which confirms the rate of increase of martensite is more than that of the twins. Microstructure examinations revealed that, the mechanisms like slip, twinning and martensite transformations occur simultaneously during the course of deformation. In crystalline materials, dislocations facilitate plastic deformation by acting as main carriers. There occurred a gradual increase in the amount of dislocations (grain interior) as a function of strain, such a dislocation activity led to the formation of twinning [12]. In a similar attempt [7] on a β titanium alloy, mechanical twinning and α" stress induced martensite were observed in the β matrix upon straining. In fact it was the evolution of phase transformation induced plasticity (TRIP) and twinning induced plasticity (TWIP) effects [7]. TWIP effect also been reported in Fe-based alloys also owing to their exceptional mechanical properties. The alloying elements in TWIP steel include Cr, Cu, N, Nb, Ti and/or V. The composition of the alloying additions influences the magnitude of the stacking-fault energy which in turn a deciding factor for mechanical properties. The stacking fault energy (γ) ranges from 15-45 mJm⁻² for TWIP steels at room temperature which activates the twinning during deformation. During the course of deformation the coordination among the glide dislocations, grain boundaries, stacking faults and twins leads to high strain hardening [13]. Stelmashenko et al. [14] stated that some classes of steel that show deformation-induced phase transformations also exhibiting good wear resistance. Christel et al. [15] summarised that, the metastable austenitic stainless steels undergo a diffusionless phase transformation from austenite to martensite by cooling or plastic deformation. This transformation is beneficial for the low cycle fatigue (LCF) properties and α'-martensite enhances the strength properties.
[15] Eskandari et al. [3] investigated the room temperature variation of strain hardening rate \( \frac{d\sigma}{d\varepsilon} \) and suggested that the high strain hardening rate is attributed to the strain-induced martensite and/or twin formation.

Rodaket al. [16] summarized that, severe plastic deformation (SPD) techniques causes microstructure refinement from macro \((1–10 \, \mu \text{m}) \rightarrow \) submicron \((300–900 \, \text{nm}) \rightarrow \) nanoscale \(<200\, \text{nm})\) grained materials. The final grain size and the mechanical properties depend on material type, deformation technique used and processing conditions. Due to the extensive use of austenitic stainless steels in industrial applications there is a need to modify the microstructure and enhance the mechanical properties. However, the mechanical properties are improved by the strain hardening and austenite phase transformation. The martensitic transformation merely depend on the stable nature of the phase (austenite) present close to or below the room temperature [16]. Nitinol used for medical applications such as manufacture of stents in peripheral and coronary bypass surgery and also in orthopedics since from late 1960s. Nitinol’s special mechanical behavior comes from its phase transformations (from austenite to martensite) occur due to coordinated atomic movements. Apart from phase transformation along composition and prior processing (i.e. rolled, drawn, extruded, or forged) also has prominent role in enhanced mechanical strength of Nitinol’s [17].

3. Conclusions
Current study is an attempt to understand phase transformations occurring in various metals like steel, titanium and super alloys. Low-carbon steels alloyed with Nb and Nb-Mo affected the microstructure and CCT diagram. The microstructure refinement in titanium alloy after THT attributed to the occurrence of phase transformation due to hydrogen induced changes in the phase components. Processing of titanium and its alloys above the \( \beta \) transus temperature leads to the formation of lamellar microstructure and the processing below the \( \beta \) transus temperature forms equiaxed microstructure. Stress-strain curve for 304 stainless steel suggested the microstructural changes accounting for deformation induced twinning and martensite transformation and the same has been reported for austenitic stainless steels NiTi alloys and others.

References
[1] B. A. Mutaftschiev (Ed), 1981, Proceedings of the Advanced Study Institute Programme, pp. 1–669.
[2] S. Suwas and R. K. Ray, 2014, Crystallographic Texture of Materials. New York: Springer.
[3] M. Eskandari, A. Zarei-Hanzaki, and A. Marandi, (2012), Mater. Des., 39 pp. 279–284.
[4] Y. L. Hao, S. J. Li, S. Y. Sun, C. Y. Zheng, Q. M. Hu, and R. Yang, (2005), Appl. Phys. Lett., 87 pp. 2003–2006.
[5] N. Isasti, D. Jorge-badiola, M. L. Taheri, and P. Uranga, 2013, Metall. Mater. Trans. A, pp. 27–38.
[6] S. Bein and J. Béchet, 1996, Le J. Phys. IV, 06 pp. C1-99-C1-108.
[7] J. Y. Zhang, J. S. Li, Z. Chen, Q. K. Meng, F. Sun, and B. L. Shen, 2017, J. Alloys Compd., 699 pp. 775–782.
[8] J. Zhao, H. Ding, Y. Zhong, and C. S. Lee, 2010, Int. J. Hydrogen Energy, 35 pp. 6448–6454.
[9] J. Chraponski and W. Szkliniarz, 2001, Mater. Charact., 46, pp. 149–154.
[10] S. Tamirisakandala, B. V Vedam, and R. B. Bhat, 2003, J. Mater. Eng. Perform., 12, pp. 661–673.
[11] Z. J. Xie, S. F. Yuan, W. H. Zhou, J. R. Yang, H. Guo, and C. J. Shang, 2014, Mater. Des., 59, pp. 193–198.
[12] Y. F. Shen, X. X. Li, X. Sun, Y. D. Wang, and L. Zuo, 2012, Mater. Sci. Eng. A, 552, pp. 514–522.
[13] B. C. De Cooman, Y. Estrin, and S. K. Kim, 2018, Acta Mater., 142, pp. 283–362.
[14] N. A. Stelmashenko, M. G. Walls, L. M. Brown, and Y. V. Milman, 1992, NATO Advanced Study Institute, 28, pp. 3–605.

[15] H.J. Christ, U. Krupp, C. Mueller-Bollenhagen, I. Roth, and M. Zimmerman, 2009, 12th International Conference on Fracture, pp. 1–10.

[16] K. Rodak, J. Pawlicki, and M. Tkocz, 2012, IOP Conf. Ser. Mater. Sci. Eng., 35.

[17] A. Saigal and M. Fonte, 2011, Mater. Sci. Eng. A, 528, pp. 5551–5559.