Improvement in the microhardness and corrosion behaviour of Ti-14Mn biomedical alloy by cold working

M K Gouda 1, Salah A Salman 1,2 and Saad Ebied 3

1 Department of Mining and Petroleum Engineering, Faculty of Engineering, Al-Azhar University, Cairo, 11371, Egypt
2 Institute of Innovation for Future Society, Nagoya University, Nagoya, Japan
3 Department of Production Engineering and Mechanical Design, Faculty of Engineering, Tanta University, Tanta, 31527, Egypt
E-mail: mohammed.gouda@azhar.edu.eg

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Abstract

β-titanium alloys are essential in many applications, particularly biomedical applications. Ti-14Mn β-type alloy was produced using an electric arc furnace from raw alloying elements in an inert atmosphere. The alloy was homogenized at 1000°C for 8 h to ensure the complete composition distribution, followed by solution treatment at 900°C, then quenched in ice water. The alloy was subjected to cold deformation via cold rolling with different ratios: 10, 30, and 90%.

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The microstructure shows a combination of twinning and slipping deformation mechanisms in the deformed alloy. Microhardness values indicated a linear increase equal to 30% by increasing the ratio of cold deformation due to the strain hardening effect. The corrosion resistance of Ti-14Mn alloy was doubled after 90% cold rolling.

1. Introduction

Titanium and titanium alloys are considered essential metallic materials. Their outstanding properties include high specific strength, high corrosion resistance, excellent biocompatibility, and appropriate mechanical properties [1, 2]. Therefore, Ti alloys are widely applicable in several applications such as the chemical industry, aerospace, automotive, and biomedical applications [3, 4].

β-titanium alloys attract the attention of many researchers as they show an exceptional combination between high specific strength, remarkable corrosion resistance, and good workability [5, 6].

β-titanium offers a replacement of the typical metallic biomaterials such as stainless steel and Ti-64. Most of these metallic biomaterials contain alloying elements harmful to the human body. Several reports indicate that alloying elements such as Al, Ni, and V could cause allergic reactions, cytotoxic effects, and neurological disorders [7].

Furthermore, most of the β-type Ti-alloys consist of high-cost rare earth elements such as Ta, Nb, and V [8, 9], limiting their possibility of replacing the traditional metallic materials. Therefore, several attempts were made to replace the high-cost toxic alloying element in the β-titanium. One of the promising attempts was using the Ti-Mn binary system [10, 11].

On the other hand, the excellent workability of β-titanium alloys, particularly cold working, allows for manipulation of alloys properties to meet the biomedical applications' requirements. Generally, cold deformation significantly impacts the deformed microstructure of alloys and, consequently, the mechanical and corrosion properties [12–14].

Corrosion resistance is vital for materials used for biomedical applications. Therefore, the effect of cold working on the corrosion resistance of Ti alloys is reported in many studies. For instance: Zhou et al [13] found that the cold rolling process does not influence the formation of passive films on the Ti-Mo alloys investigated in
The β divided into cuboid-shaped specimens with dimensions of 5 × 0.5 × 0.5 cm for (Length × width × Thickness) by wire electrical discharge machining (Brother HS300).

X-ray diffraction (XRD) technique was used to examine the existing phases by (XPERT MPD, Philips, Japan) diffractometer with Ni-filtered Cu Kα operated at 30 kV and 30 mA in a 2θ range of 30–80 degrees. Microstructure investigations were carried out using Field Emission Emission Microscopy (FESEM) after the standard grinding and polishing procedure was followed. A four-high rolling mill was used to apply cold working while maintaining the rolling direction constant and without intermediate annealing.

The mechanical behaviour was evaluated using the Microhardness technique, each specimen was measured 15 times, and the mean is considered the micro-hardness value. A conventional three-electrodes cell configuration was used to assess corrosion behaviour. The reference electrode was Ag/AgCl, platinum foil, and the specimens were used as the counter and working electrodes, respectively. Ringer solution (sodium chloride, potassium chloride, calcium chloride, and sodium bicarbonate in the concentrations in which they occur in body fluids) was used as an electrolyte at a fixed temperature of 37 °C.

3. Results and discussion

Actual compositions of the Ti-14Mn alloy, referred to by (14Mn) alloy hereafter, are shown in Table 1.

| Sample name | Mn | O  | N  | Ti  |
|-------------|----|----|----|-----|
| (14Mn) alloy | 14.1 | 0.12 | 0.0050 | Bal. |

5% HCl solution at 310 K. Moreover, Guo et al. [15] concluded that the corrosion resistance of Ti-6Al-3Nb-2Zr-1Mo alloy increases with the increase of cold rolling deformation owing to the grain refining and texturing after cold rolling.

However, the studies of the effect of cold deformation on the Ti-Mn binary system are limited; therefore, in the current study, Ti1–4Mn were produced, homogenized, solution treated, and cold deformed via cold rolling with different ratios 10, 30, and 90%. The phases change, microstructure, mechanical properties, and corrosion resistance of Ti-14Mn alloy were evaluated before and after cold rolling.

2. Methods

The current study used a sealed electric arc furnace (ARCAST 200, USA, 200 gm crucible with vacuum or inert gas) to produce the alloys under an argon atmosphere. Homogenization was done at 1000 °C for 8 h using encapsulated sealed argon gas-filled quartz tube inside a muffle furnace to ensure the complete composition distribution. After homogenization, solution heat treatment at 900 °C for 30 min was applied, followed by quenching in ice water to produce a complete single β-phase. Samples were produced in the ingot shape then divided into cuboid-shaped specimens with dimensions of 5 × 0.5 × 0.5 cm for (Length × width × Thickness) by wire electrical discharge machining (Brother HS300).

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Inductively coupled plasma - optical emission spectrometry (ICP-OES) technique was used to verify the actual composition combined with Helium carrier fusion infrared adsorption method for oxygen and Helium carrier fusion thermal conductivity for Nitrogen. The Oxygen content is a little bit high; however, it is still in the allowable range. Furthermore, the nitrogen content is low and can be negligible.

3.1. Phases and microstructure change

The effect of cold rolling on the existed phases and morphology was investigated by XRD and SEM techniques.

Figure 1 presents the XRD patterns for the studied alloys before cold rolling (ST) and after cold rolling (CR). β-phase is the only detectable phase in the ST condition, and there are no peaks for other phases such as α, α’, ω or intermetallic compounds in (14Mn) alloy. This means β-phase is the only existed phase after quenching from the β-phase zone at 14Mn wt.% in the current study.

It is widely known that titanium alloying elements are classified into neutral, α-stabilizers, and β-stabilizers [16–18]. Elements that expand the α-phase field to higher temperatures are referred to as α-stabilizing elements. On the other hand, elements that move the β-phase field to lower temperatures are referred to as β-stabilizing elements, whereas neutral elements do not affect the β-transus temperature.

There are two types of β-stabilizing elements: isomorphous and eutectoid. The isomorphous type has complete solid solubility. In contrast, the low solubility and tendency for intermetallic compound formation are
Mn is a strong eutectoid-type $\beta$-stabilizing element that stabilizes the $\beta$-phase to be more dominant even at room temperature. This means that 14% Mn-content is sufficient to retain all $\beta$-phase after solution treatment, in a complete consensus with the previous reports, indicating that 7% Mn is adequate to produce a stable single $\beta$-phase in the Ti-Mn binary alloys [21, 22].

Moreover, after cold rolling, the existence of the $\beta$-phase remained constantly from a low degree of deformation (10%CR) to a high degree (90%CR). There are no signs of stress-induced transformation even at a high degree of deformation when severe plastic deformation generated by the cold rolling process is expected. The generation of the stress-induced transformation was reported in Ti-Mn binary alloys; however, it was at a lower concentration than 14Mn [11].

The Field Emission Scanning Electron Images (figure 2) show no signs for any phase other than the $\beta$-phase, which agrees with the XRD results in figure 1. Furthermore, (14 Mn) alloy shows large equiaxial single $\beta$-phase grains ranging between 150 to 500 $\mu$m and other phases and intermetallic compounds are not visible, as shown in figure 2(a).

In addition, slipping deformation mechanisms are visible at a low degree of deformation (10%CR) with a few twins, as shown in figure 2(b).

Slip or slipping is the most common mechanism of metal plastic deformation. It involves sliding crystal blocks over one another along defined crystallographic planes [23]. Twinning is simply a movement of planes of atoms in the lattice parallel to a specific plane (twinning plane), so the lattice is divided into two symmetrical parts that are differently oriented [24]. Both mechanisms were reported during the deformation of titanium and titanium alloys [25–28].

By increasing the degree of deformation (30%), the co-occurrence of slipping and winning deformation mechanisms is more evident than the low degree of deformation with the appearance of the grain subdivision [29], as shown in figure 2(c). At a high degree of deformation (90%), grains rotate and align parallel to the direction of rolling that forms a rolling texture (lamellar structure). The \{100\}$_{\beta}$ (100) rolling texture is generally formed after cold rolling due to the dislocation movement and grains rotation, as discussed in various literature [9, 29–32]. In this texture, the \{200\}$_{\beta}$-planes aligned parallel to the rolling plane. Therefore, the measured x-ray peak intensity ratio of the cold-rolled specimen, IR$_{cr}$, changed with cold rolling. IR$_{cr}$ is defined as the ratio between the x-ray peak intensities of the [200]$_{\beta}$ and [110]$_{\beta}$ reflections. In the current study, the IR$_{cr}$ seems to increase with increasing the degree of deformation in (14Mn) alloy, as shown in figure 1 [32].

### 3.2. Microhardness test results

As an initial assessment of the strength, microhardness tests were applied at a load equal to 0.5 Kg, and the time of indentation was 15 seconds. Each specimen was measured 15 times, and the mean was introduced here as the
microhardness value, as shown in figure 3. As can be noticed in this figure, the micro-hardness values increase monotonously with the increase in the cold rolling ratio. The microhardness value increases from 390 in the ST condition to 410, 435, 485 HV0.5 in 10%, 30%, and 90% CR, respectively.

The microhardness values shown in figure 3 imply that increasing the degree of deformation will result in an increase in microhardness values. This is the expected behaviour as strain hardening is a well-known phenomenon. Applying plastic deformation will lead to a generation of new dislocations, which increase the dislocation density. This means that dislocations will accumulate and interact with each other and act as obstacles. Consequently, the dislocation motion will impede results in an increase in strength.
3.3. Corrosion behaviour

Potentiodynamic polarization techniques examined the corrosion resistance of 14Mn in both ST and CR conditions. The experiment was performed in Ringer’s solution at 37 °C to simulate an environment near human body fluids. Tafel plots were used to extract corrosion potential values (E_{corr}) and corrosion current density (I_{corr}). These values were used to assess the anti-corrosion property of the studied alloys before and after cold rolling.

The potentiodynamic polarization curves of (14Mn) alloy in ST, 10% CR, 30% CR, and 90%CR conditions were presented in figure 4. This figure shows that all alloys show the same linear behaviour with a clear passive nature owing to the formation of a spontaneous protective passive film, mainly from TiO_2, on the surface of the studied alloys as previously reported in titanium and titanium alloys [13, 33–37]. The essential corrosion parameters (I_{corr} and E_{corr}) are concluded from the potentiodynamic polarization curves, corrosion rate was calculated using the equation mentioned below [38], and the results are listed in table 2. I_{corr} is the corrosion current density (μA/cm²), ρ is the alloy’s density (g/cm³), and EW is equivalent to weight.

Values of (E_{corr}) are barely changing, possibly because of the existence of a single β-phase. However, it is known that I_{corr} can provide a more accurate indication of the corrosion rate than E_{corr} [39, 40]. I_{corr} is decreased by increasing the degree of deformation, and the lowest I_{corr} value was recorded at the highest degree of deformation (90%CR) in the current study.

Furthermore, the potentiodynamic polarization curves presented in figure 4 suggest that cold rolling increases the corrosion resistance of (14Mn) alloy. As shown in this figure, (14Mn) alloy in the solution treated condition shows higher passive current density higher than cold rolled condition. Moreover, by increasing the degree of deformation, the passive current density decreases, implying that cold rolling increase the corrosion resistance of (14Mn) alloy.

In general, cold deformation has a considerable impact on the microstructure of deformed alloys and, as a result, on their corrosion properties [10, 19, 41]. The active sites on a metal’s surface, such as grain boundaries and dislocations, are beneficial to passive layer formation and substantially impact the passivation process [40]. It is strongly believed that the grain refinement and high dislocation density created by cold rolling provides more nucleation passivation sites, increasing the corrosion resistance of cold-rolled alloys.

The effects of deformation on corrosion resistance have already been reported; Balyanov et al [42] indicated that the passive surface formed easily in ultra-fine grains caused by the ECAP technique compared to coarse-

| Sample     | I_{corr} (μA) | E_{corr} (mV) | Corrosion Rate (Mm/Year) |
|------------|--------------|--------------|--------------------------|
| 14Mn ST    | 9.60         | 200          | 0.784 × 10^{-4}          |
| 14Mn 10% CR| 7.34         | 189          | 0.600 × 10^{-4}          |
| 14Mn 30% CR| 5.30         | 185          | 0.432 × 10^{-4}          |
| 14Mn 90% CR| 0.055        | 177          | 0.044 × 10^{-4}          |

EW = 12.214235 gm and ρ = 4.892 g cm⁻³

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grained grains in titanium alloys. Kumar et al [43] argued that the grain refinement of T-64 alloy by ultrasonic shot peening process is the main reason for improving corrosion resistance. Moreover, rolling texturing formed during the cold rolling process seems to affect the corrosion resistance of (14Mn) alloy positively. Similar behaviour was reported in steel alloys when the corrosion resistance of stainless steel alloy improved by forming close pack crystallographic planes parallel to the specimen surface (texturing) [14]. Moreover, the values of corrosion potential $E_{\text{corr}}$ and $I_{\text{corr}}$ values indicate that applying plastic deformation (cold rolling) causes a significant improvement in the corrosion resistance of (14Mn) alloy.

4. Conclusions

The phases change, microstructure, mechanical properties, and corrosion resistance of Ti-14Mn alloys were evaluated before and after cold rolling. $\beta$-phase is the only existed phase even after a high degree of deformation. Deformation mechanisms were slipping and twinning during the cold deformation Ti-14Mn alloy. It was found that the microhardness increases with increasing the degree of cold rolling. The degree of cold rolling affects the corrosion resistance of Ti-14Mn alloy. The highest corrosion resistance was obtained at 90% CR.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

ORCID iDs

M K Gouda @ https://orcid.org/0000-0003-1326-9706
Salah A Salman @ https://orcid.org/0000-0002-1740-9983

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