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IMPROVEMENT OF MECHANICAL PROPERTIES FOR SOME Ti-Mo-Fe ALLOYS BY HEAT TREATMENT

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The mechanical properties of Ti-Mo-Fe alloys were enhanced by applying a specific heat treatment. Five alloy samples with various Fe concentrations (1%, 2%, 3%, 4%, and 5%), were subjected to optical (OM) and scanning electron microscopy - energy-dispersive spectroscopy (SEM-EDS) analysis. The heat treatment involved quenching at 750˚C for 60 minutes followed by water cooling, and a subsequent ageing heat treatment at 550˚C with two different holding times, 240 minutes followed by water cooling, and 360 minutes followed by water cooling. The results of the study showed improved mechanical properties.

Keywords: quenching, ageing, optical microscopy, scanning electron microscopy, microhardness test, Tiβ alloy.

1. Introduction

Ti-Mo-Fe alloys exhibiting mostly β structure have several advantages as low-cost biocompatible materials. One of the main advantages is their excellent mechanical properties. For example, porous Ti - 10Mo - xFe alloys have a compressive yield strength of 500 to 800 MPa, much higher than that of human bone (about 130 - 180 MPa) [1]. Additionally, these alloys have a low elastic modulus (< 10 GPa), which is closer to that of human bone (10 - 30 GPa), than other metallic biomaterials such as pure titanium (110 GPa) [2]. This can help reduce the stress shielding effect that can occur when there is a mismatch between the elastic modulus of the implant and the surrounding bone tissue. Ti-Mo-Fe alloys

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also have good corrosion resistance, making them suitable for use in medical implants [3]. This paper explores the impact of different heat treatments on the microstructure and mechanical properties of Ti-Mo-Fe alloys.

There are studies on the use of Ti-Mo-Fe alloys as biocompatible alloys. One study found that porous Ti–10Mo–xFe (x = 2–5) alloys prepared by powder metallurgy using ammonium hydrocarbonate (NH_4HCO_3) as space-holder have superior mechanical properties, good corrosion resistance, and excellent biocompatibility, making them promising candidates for bone substitute materials [4]. Another study compared the microstructural evolution and strengthening mechanisms in two biocompatible alloy systems, the binary Ti-15Mo and the quaternary Ti-13Mo-7Zr-3Fe (TMZF) [5]. The TiMoFe alloy, with its biocompatibility due to titanium, deformability conferred by 15% molybdenum, improved mechanical properties and cost reduction from 1-5% iron, holds significant potential for medical applications.

Heat treatment of Ti-Mo-Fe alloys involves several steps, including quenching and ageing. For example, one study found that heat treatment at 900°C for 10 hours increased the corrosion resistance of as-cast alloys regardless of the Ti content [6].

2. Experimental technique

Heat treatments were performed in a Nabertherm HTC 03/14 induction furnace, of 9 kW and 50/60 Hz frequency that reaches temperatures up to 1400 °C.

To enhance the hardness of Ti-Mo-Fe system alloys, 10 samples were treated in the Nabertherm 03/14 heat treatment furnace at 750°C for 1 hour followed by water cooling and submitted to ageing heat treatment at 550˚C with two different holding times, 5 samples for 240 minutes followed by water cooling, and 5 samples for 360 minutes followed by water cooling.

The purpose of rapid cooling subsequent to ageing is to prevent the precipitation of metastable ω phase, which causes embrittlement. These phases diminish mechanical performance, including tensile strength, making the achievement of a predominantly β structure essential [7].

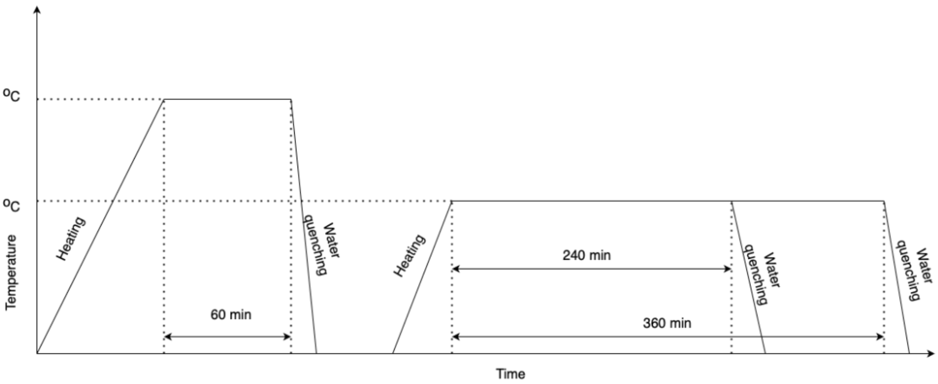
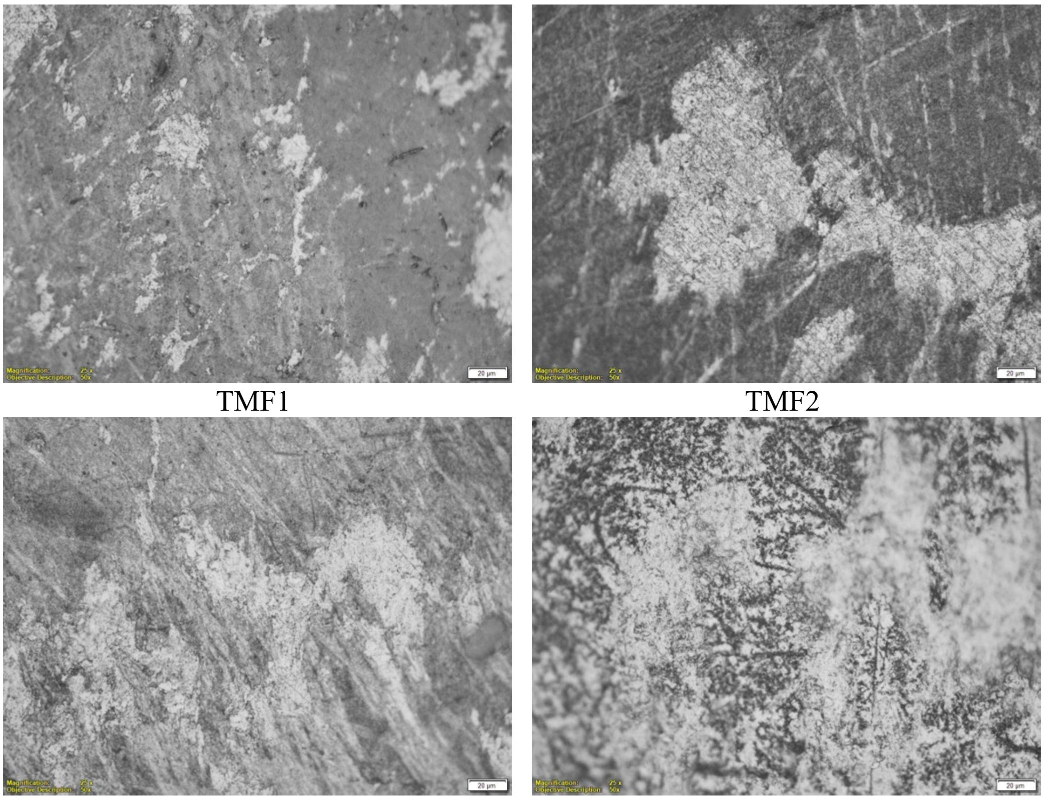


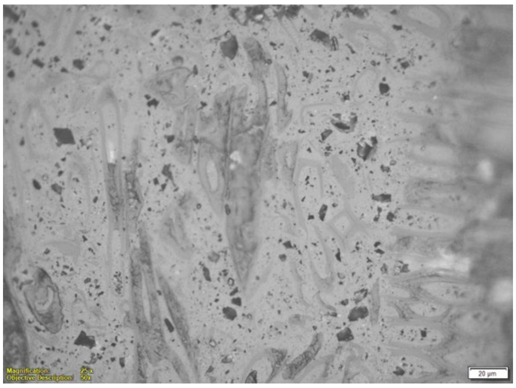
Fig. 1. Quenching and ageing heat treatments diagram.

The samples were tested by applying a force of 300 gf for 15 seconds. Microstructural examination was performed on Olympus BX60M optical microscope. Samples were prepared and etched with Kroll's reagent.



TMF1 TMF2

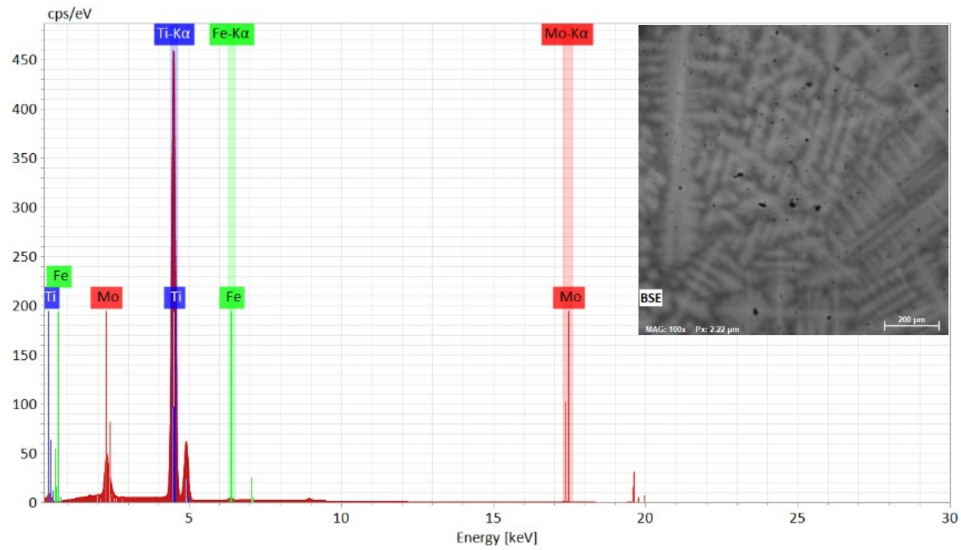
TMF3 TMF4



TMF5

Fig. 2. Optical microscopy analysis of TMF samples without heat treatment.

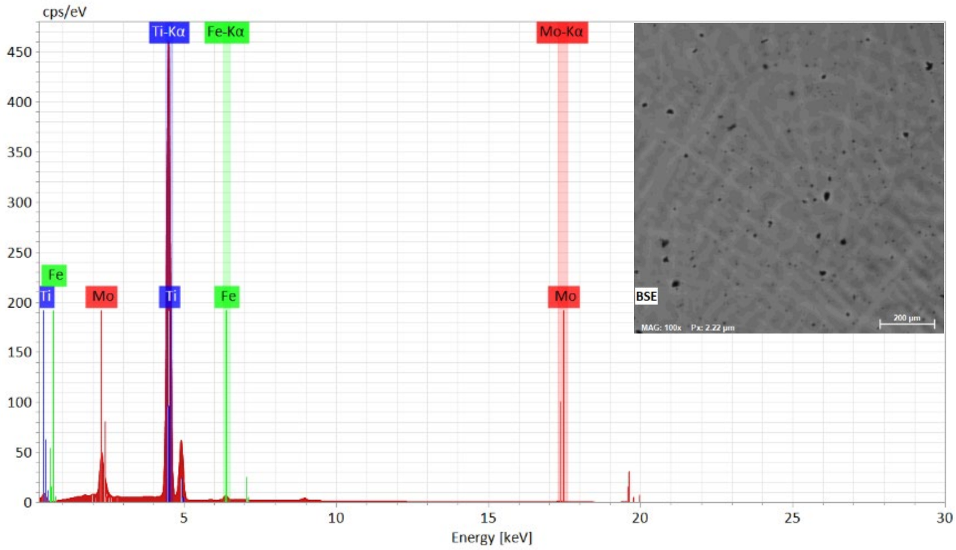
Additionally, the samples were analysed using a TESCAN VEGA II – XMU scanning electron microscope (SEM) fitted with a Bruker Quantax XFlash 630M EDS detector, in order to reveal structural characteristics and perform elemental analysis, as shown in Figure 3.



Map.xlsx

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Titanium	22	161440066	67.23	89.51	94.34	1.86	2.77
Molybdenum	42	642993	7.59	10.11	5.32	0.23	3.01
Iron	26	464154	0.29	0.38	0.34	0.03	11.25
		Sum	**75.11**	**100.00**	**100.00**		

TMF1



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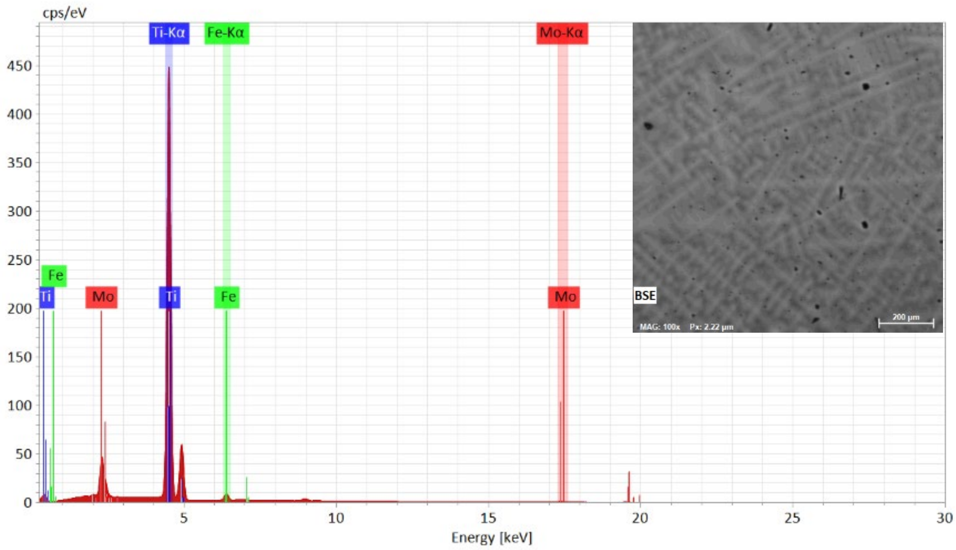
Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Titanium	22	160931343	66.75	88.93	93.78	1.85	2.77
Molybdenum	42	634225	7.52	10.02	5.27	0.23	3.01
Iron	26	1281568	0.79	1.05	0.95	0.04	5.69
		Sum	**75.05**	**100.00**	**100.00**		

TMF2



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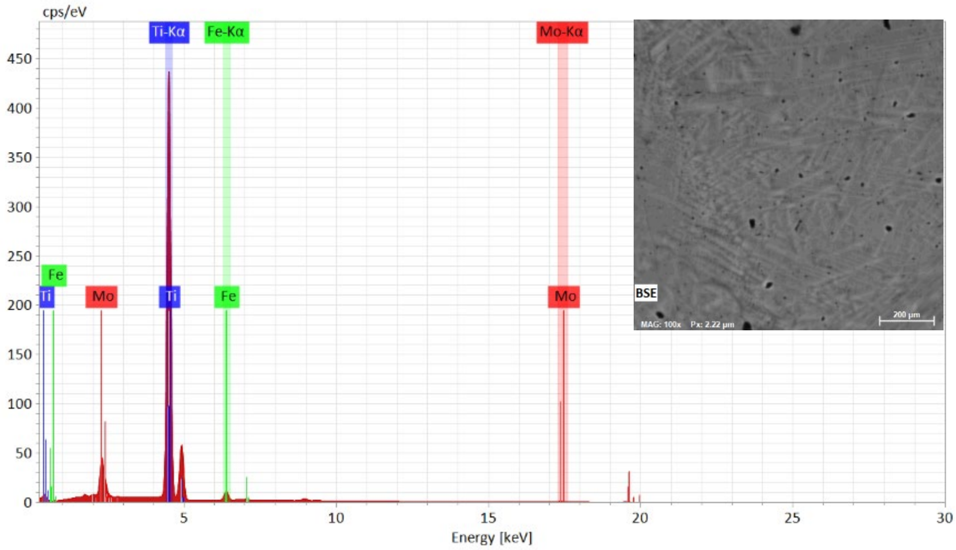
Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Titanium	22	155836224	66.09	87.76	92.86	1.83	2.77
Molybdenum	42	652587	7.86	10.44	5.51	0.24	3.00
Iron	26	2160441	1.36	1.80	1.63	0.06	4.36
		Sum	**75.31**	**100.00**	**100.00**		

TMF3



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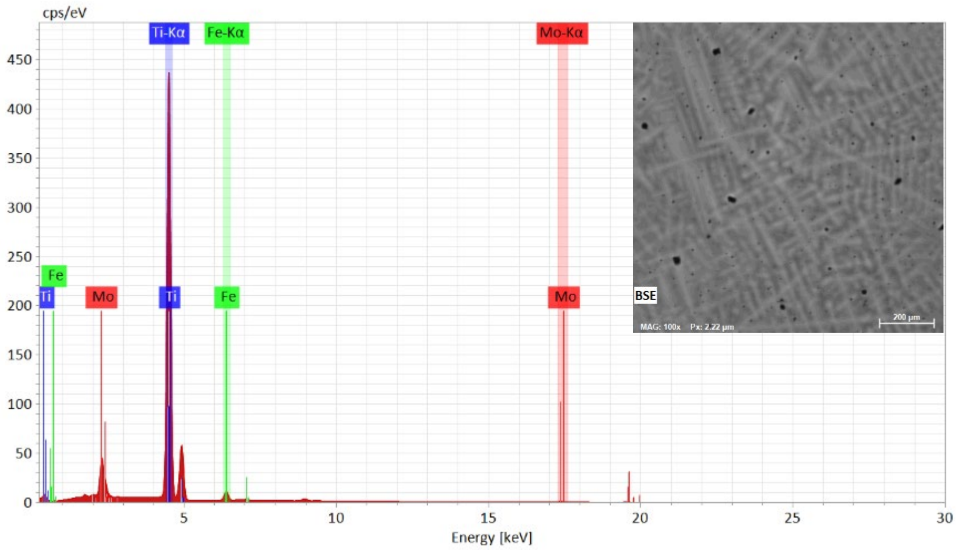
Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Titanium	22	151900692	65.03	87.36	92.37	1.80	2.77
Molybdenum	42	624753	7.52	10.11	5.33	0.23	3.01
Iron	26	2971751	1.88	2.53	2.29	0.07	3.85
		Sum	**74.44**	**100.00**	**100.00**		

TMF4



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Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Titanium	22	154945077	65.27	87.14	91.99	1.81	2.77
Molybdenum	42	586616	7.18	9.59	5.05	0.22	3.03
Iron	26	3925635	2.45	3.27	2.96	0.09	3.54
		Sum	74.91	100.00	100.00		

TMF5

Fig. 3. SEM-EDS characterization of TMF1-5 alloys quenched samples at 750°C/ 1h highlighting the α" + β structure

All samples are showing a dendritic microstructure and for all specimens, Ti, Mo and Fe were indexed in the obtained spectrum. Analysing the data presented in fig. 3.3, one can observe that the Fe content estimated by means of SEM-EDS analysis for the examined microstructural fields is gradually increasing from sample TMF1 to TMF5 (see Mass Norm., %), showing a good Fe concentration consistency for the investigated alloys.

For the microhardness tests, 5 different indentations were performed to obtain the average values.

In Tables 1 ÷ 4 and Figures 4 ÷ 7, the average microhardness values and their evolution following quenching and ageing heat treatments for Ti-Mo-Fe alloys are presented.

Table 1

HV microhardness values of TiMoFe samples without heat treatment

	1%Fe	2%Fe	3%Fe	4%Fe	5%Fe
Average	415	498	412	579	676

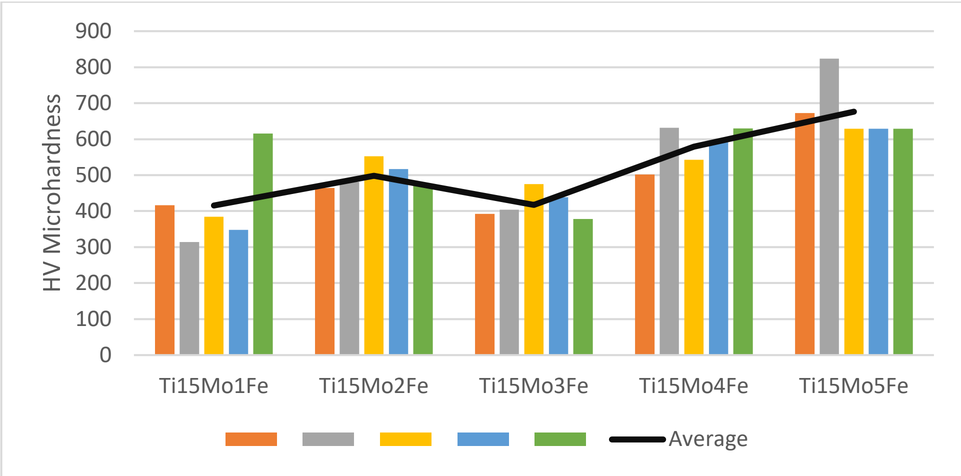

Fig. 4. Plots of HV microhardness test values for samples without heat treatment

Table 2

HV microhardness values of TiMoFe samples after quenching at 750°C / 60 min/ water

	1%Fe	2%Fe	3%Fe	4%Fe	5%Fe
Average	480	624	570	502	530

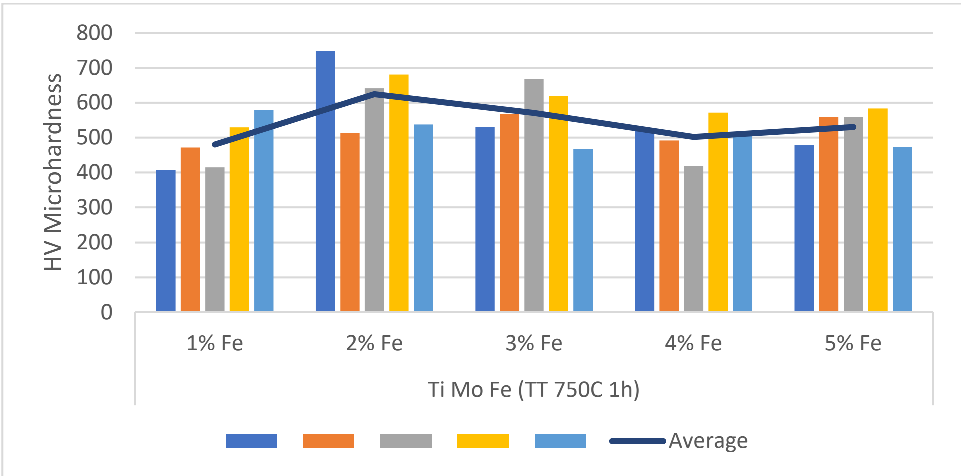

Figure 5. Plots of HV microhardness test values for samples heat treated at 750°C/ 60 min/water

Table 3

HV microhardness values of TiMoFe samples after ageing at 550°C/ 240 min/ water

	1%Fe	2%Fe	3%Fe	4%Fe	5%Fe
Average	384	385	455	461	466

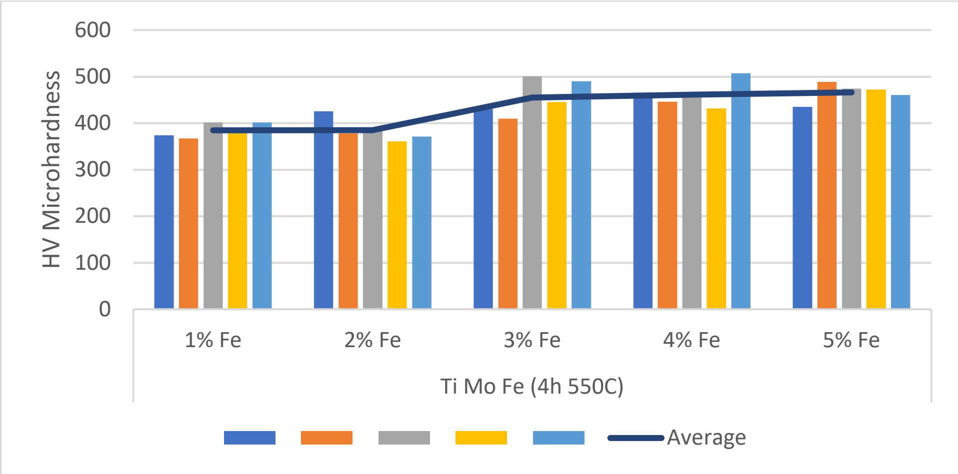

Fig. 6. Plots of HV microhardness test values for samples aged at 550°C/ 240 min/ water

Table 4

HV microhardness values of TiMoFe samples after artificial ageing at 550°C/ 360 min/ water

	1%Fe	2%Fe	3%Fe	4%Fe	5%Fe
Average	429	467	495	456	467

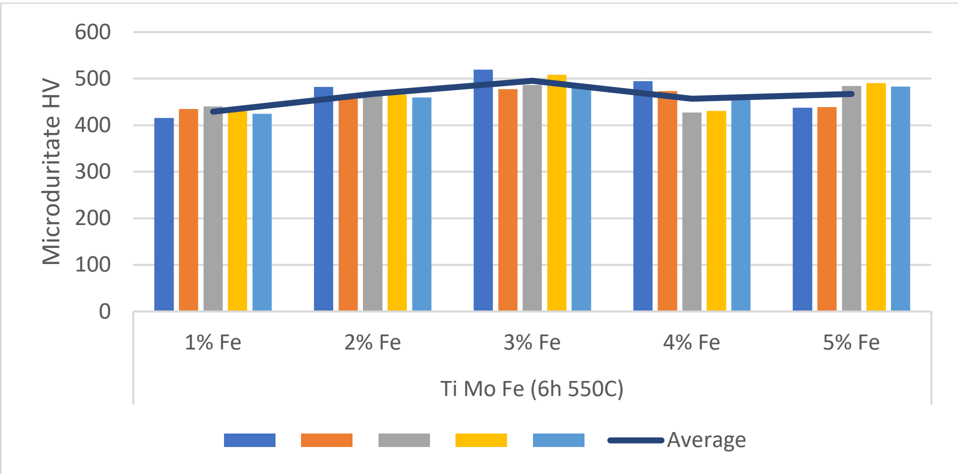

Fig. 7. Plots of HV microhardness test values for samples aged at 550°C/ 360 min/ water

The analysis reveals that specific heat treatments, comprising quenching at 750 °C followed by water cooling and ageing at 550 °C (for holding times of 240 and 360 minutes, with water cooling), result in an enhancement of the microhardness values across all tested samples as can be seen in Table 5 and Figure 8. This establishes a direct correlation between the applied heat treatments and the improved mechanical properties of the alloys.

Moreover, the data demonstrates a tendency for microhardness to raise when increasing the alloys Fe concentration.

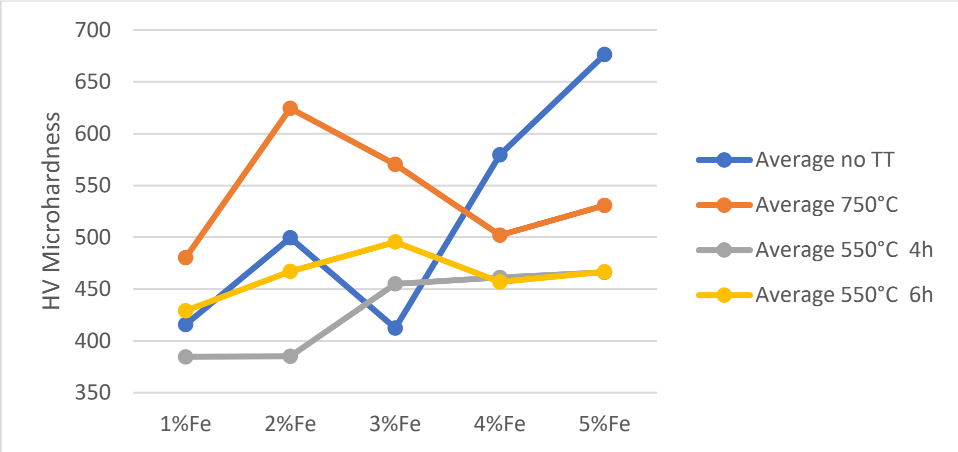

Fig. 8. Graphs depicting the average values for all samples.

Table 5

HV microhardness values of TMF for all samples

	HV Microhardness values				
	TMF1	TMF2	TMF3	TMF4	TMF5
Average no TT	415	499	412	579	676
Average 750°C	480	624	570	502	530
Average 550°C/ 4h	384	385	455	461	466
Average 550°C/ 6h	429	467	495	456	467

For the samples without heat treatment, increasing the Fe concentration results in the formation of intermetallic compounds found at the grain boundaries, leading to an increase in microhardness values.

A decrease in microhardness values is observed at various Fe concentrations of the alloy, confirming the findings of E. Awannegbe et. Al. [7] due to the dissolution of brittle intermetallic compounds.

TMF1 exhibits an over 15% increase from no heat treatment to quenching, a 20% decrease from quenching to 4 hours of ageing, and an over 3% increase from 4 hours to 6 hours of ageing. TMF2 shows a 25% increase from no heat treatment to quenching, an over 38% decrease from quenching to 4 hours of ageing, and an over 21% increase from 4 hours to 6 hours of ageing. TMF3 experiences an over 38% increase from no heat treatment to quenching, an over 20% decrease from quenching to 4 hours of ageing, and an over 8% increase from 4 hours to 6 hours of ageing. TMF4 demonstrates an over 13% decrease from no heat treatment to quenching, an over 8% decrease from quenching to 4 hours of ageing, and a 1% decrease from 4 hours to 6 hours of ageing. TMF5 reveals an over 21% decrease from no heat treatment to quenching, an over 12% decrease from quenching to 4 hours of ageing, and a slight increase from 4 hours to 6 hours of ageing.

Variations in microhardness values among TMF1 to TMF5 highlight the impact of Fe concentration on alloy hardness. Generally, alloys with higher Fe content exhibit higher microhardness values, underlining the Fe role in the alloy strengthening, possibly through the formation of intermetallic compounds or influencing microstructural characteristics.

Specific observations include a marked increase in microhardness values following the quenching process at 750°C for 60 minutes, evidencing the effectiveness of this heat treatment step. Following to ageing at 550°C for 240 minutes, there was a slight raise in hardness, although not as significant as after quenching for some Fe concentrations. The ageing at 550°C for 360 minutes further positively improved microhardness, showing that prolonged ageing can improve the mechanical properties of the alloys. This fact is due to intermetallic compounds intense precipitation.

3. Conclusions

The article represents a study on the mechanical properties of Ti-Mo-Fe alloys, using a specific heat treatment. A number of 10 alloy samples with different Fe concentrations (1%, 2%, 3%, 4%, and 5%) were tested by optical and electron microscopy analysis. Heat treatment involved a quenching step at 750˚C for 60 minutes, followed by water cooling and subsequent ageing heat treatment at 550˚C with two different holding times, 240 minutes followed by water cooling and 360 minutes followed by water cooling.

Rapid cooling during the ageing process is important for inhibiting the formation of ω metastable phase, which are known to cause embrittlement. Achieving a predominantly β phase structure is essential for maintaining the material performance.

It was found that the applied heat treatment led to the increase of microhardness in the case of all the samples, which highlights an improvement of the mechanical properties. It was also observed that Fe concentration had an impact on rising microhardness.

This improvement is attributed to the microstructural changes caused by the heat treatment, which aligns with the goal of improving the mechanical performance of these alloys for potential applications, particularly in contexts where biocompatibility and mechanical strength are critical, such as medical implants.

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