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MANUFACTURING NEW Fe-ALLOYED Ti-Mo ALLOYS FOR BIOMEDICAL APPLICATIONS

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Ti-Mo alloys have attracted great interest because of their biocompatibility, strength, and wear resistance. Arc melting is a common fabrication technique for these alloys, as it provides homogeneous compositions with well-controlled microstructures. The alloying elements can be introduced into the melt by using pre-alloyed powders or elemental metal rods. Biocompatibility of these alloys is ensured through the use of low levels of Mo, a known non-toxic element, alloyed with titanium.

Materials based on Ti15MoFe can be a promising candidate for orthopaedic implant applications. The alloys were studied using optical microscopy, scanning electron microscopy, energy-dispersive X-ray spectroscopy, and microhardness testing.

Keywords: Biocompatibility, Arc melting, SEM, EDS, Microhardness

1. Introduction

Titanium and its alloys are currently widely used as orthopaedic implants due to their excellent biocompatibility, mechanical, physical, and biological performance compared to other metallic biomaterials [1]. The most commonly used titanium-based material for medical applications has been Ti-6Al-4V ELI alloy, according to Yasser et al [2], who have identified new Fe-alloyed Ti-Mo alloys as promising candidates for improved mechanical properties, biocompatibility, and corrosion resistance [3].

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Fe-alloyed Ti-Mo alloys are an exciting new class of biomedical materials that offer many advantages over existing titanium alloys such as increased ductility, better surface passivation, improved wear resistance, and enhanced corrosion protection. In addition to these performance benefits, Fe-alloyed Ti-Mo alloys are also cost-effective, making them ideal for medical device applications.

Min et al. [4] mentioned that in order to obtain the full β phase in the Ti-Mo binary alloy, a minimum of 10% Mo is required to prevent martensitic transformation after quenching to ambient temperature.

At 882°C, titanium undergoes an allotropic transformation from an α-phase hcp structure to a β-phase bcc structure [5]. Titanium alloys are thus classified into three types as a result of this structural change: α alloys, α + β alloys, and β alloys. [6]. These metallographic changes can be enhanced by adding specific or alloying stabilizers [7].

a. Biocompatible Ti-based alloys

Pure titanium and some of its alloys exhibit good mechanical and chemical properties for biomaterials; however, newly developed Ti-based biomaterials prioritize material safety over physical properties [8].

According to Spătaru et al., the alloying elements that are non-toxic and non-allergenic based on cell survival for pure metals, polarization resistance, and tissue compatibility are: Si, Nb, Ta, Zr, Sn, Mo, Fe, and Hf [9].

Corrosion resistance is an essential characteristic of titanium and its alloys, due to its thin, compact and highly stable film of titanium dioxide that forms within seconds of contact with the environment [10]. Thus, due to its very good resistance to corrosion, titanium is used in medicine, it resists water, acids or salty solutions, having a behaviour comparable to platinum in the case of chemical corrosion.

b. Biocompatible Ti-Mo Alloys

Due to their high specific strength, superior corrosion resistance, and acceptable biocompatibility, β-Ti alloys have received the greatest attention in recent investigations in metal biomaterials for load-bearing implants [11].

Non-toxic β-stabilisers such as Mo, Sn, Ta, Nb, and Zr are used primarily as alloying elements to create new beta-type titanium alloys with low elastic modulus, such as Ti15Mo [12].

The following issues were considered when determining the appropriate melting process to obtain these alloys: - The alloys contain alloying elements with high melting temperatures (Ti-1668^{o}C, Mo-2896^{o}C, Fe-1538^{o}C), necessitating a temperature minimum obtained in the melting furnace. The vacuum arc furnace used achieves temperatures of 3700^{o}C.; - large differences in melting temperatures and density can create segregation problems. No such defects were encountered since Mo and Fe dissolve easily in titanium (the equilibrium diagrams show the existence of broad β solid solution, domains that widen with increasing Mo or Fe content) (Fig.. 1.1).

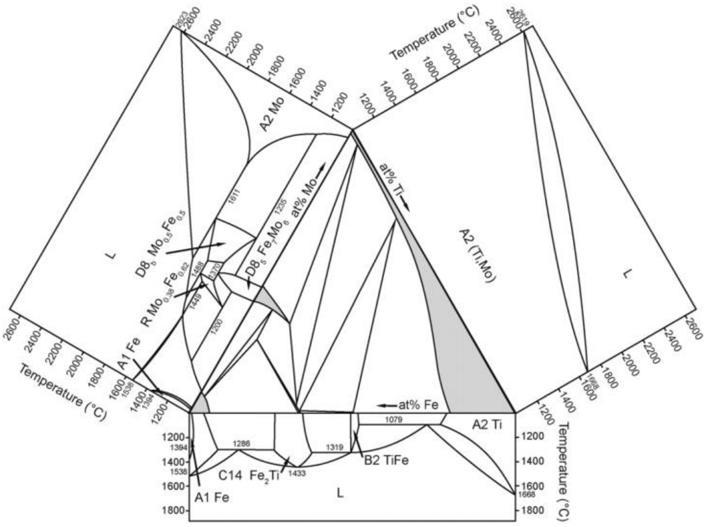


Fig.. 1.1. Binary Fe-Ti, Fe-Mo and Mo-Ti phase diagrams and Fe-Mo-Ti ternary phase diagram [13]

2. Experimental technique

For the experimental procedure, the MRF ABJ 900 vacuum arc remelting furnace (Fig. 2.1) which must ensure the ability to melt (obtain) metallic materials in an argon-controlled atmosphere after prior vacuuming of the work area up to 10^{-6} bar, using an electric arc formed by a non-consumable tungsten thoriated mobile electrode and the base plate.

Fig.. 2.1. ABJ MRF 900 furnace and interchangeable Cu crucible

The MRF ABJ 900 vacuum arc remelting installation allows the melting of metals with high melting points such as Mo, Zr, W, Co, Ni, etc., thus creating the possibility of obtaining the most diverse metal alloys. Exceptions are alloys containing metals with a low vaporization temperature, such as Mg, Zn, etc.

These alloys were created by arc melting in a vacuum, a process that ensured good homogeneity due to intense agitation and the elimination of impurities through evaporation due to the advanced vacuum (10^{-6} bar).

Because of the intense stirring and short cooling time, the process also ensures that the structure has advanced finesse in terms of the shape and size of the crystalline grains. The very short cooling time - processing is done in a cold crucible - also ensures the obtaining of a single-phase β structure, stable structure above 850^{o}C, no longer requiring a solution treatment or stress relief. Five samples with different wt% concentrations of Fe were fabricated for the experiments, as shown in Table 2.1. The quantities of pure metals used to obtain the alloys are shown in Table 2.2.

Table 2.1

Alloy mass calculation

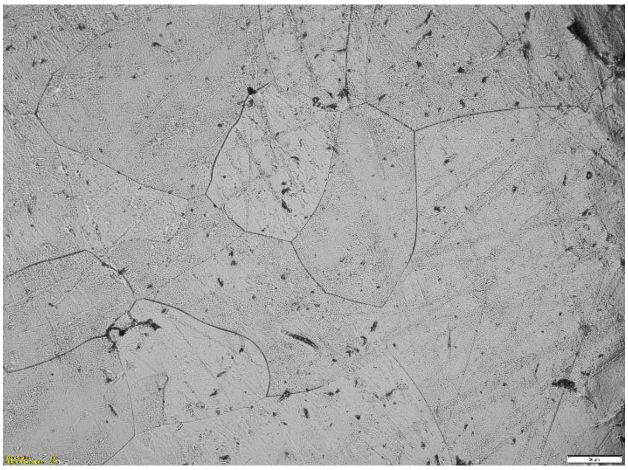
Mo density (g/cm^3)	Ti density (g/cm^3)	Fe density (g/cm^3)	%Mo	%Ti	%Fe	Total (%)	Alloy density (g/cm^3)	Estimated alloy volume (cm^3)	Calculated alloy mass (g)
10.3	4.5	7.9	15	84	1	100	5.40	5.50	29.72
10.3	4.5	7.9	15	83	2	100	5.44	5.50	29.91
10.3	4.5	7.9	15	82	3	100	5.47	5.50	30.10
10.3	4.5	7.9	15	81	4	100	5.51	5.50	30.28
10.3	4.5	7.9	15	80	5	100	5.54	5.50	30.47

Table 2.2

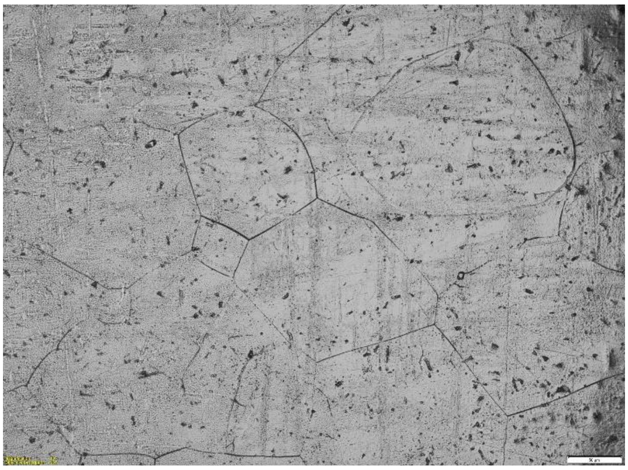
Total alloy mass per alloying element

Sample	% Mo	Grams Mo	% Ti	Grams Ti	% Fe	Grams Fe	Total alloy mass (g)
Ti15Mo1Fe	15	4.46	84	24.97	1	0.30	29.72
Ti15Mo2Fe	15	4.49	83	24.82	2	0.60	29.91
Ti15Mo3Fe	15	4.51	82	24.68	3	0.90	30.10
Ti15Mo4Fe	15	4.54	81	24.53	4	1.21	30.28
Ti15Mo5Fe	15	4.57	80	24.38	5	1.52	30.47

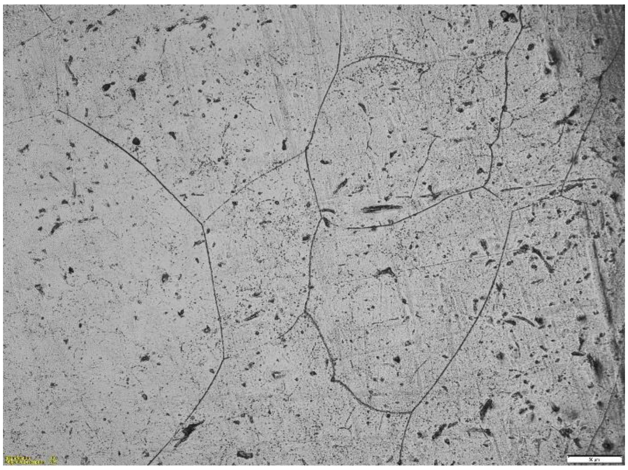
Keller's reagent was used to etch the samples in order to prepare them for the MO analysis, the solution contains 190 ml of distilled water, 5 ml of nitric acid, 3 ml of hydrochloric acid, and 2 ml of hydrofluoric acid. An immersion time of 10-30 seconds is required to visibly etch the material. The reagent is ideal for aluminium and titanium alloys. Fig. 2.2 shows optical micrographs of the samples containing 1, 2, 3, 4, and 5% Fe at x200 magnification, the images were taken with an Olympus BX51M microscope equipped with an Olympus UC30 camera system.



Ti15Mo1Fe x200

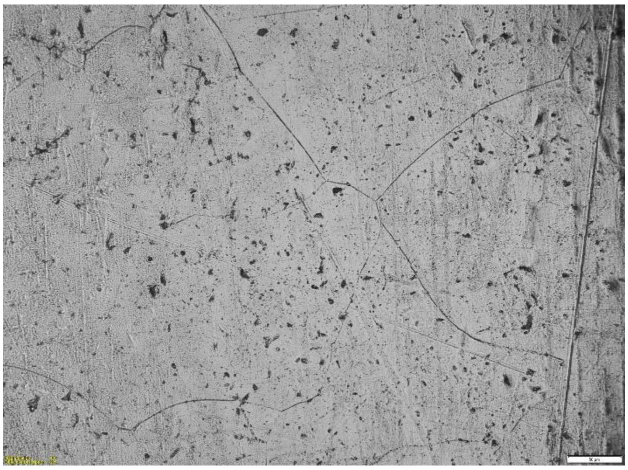


Ti15Mo2Fe x200



Ti15Mo3Fe x200

Ti15Mo4Fe x200

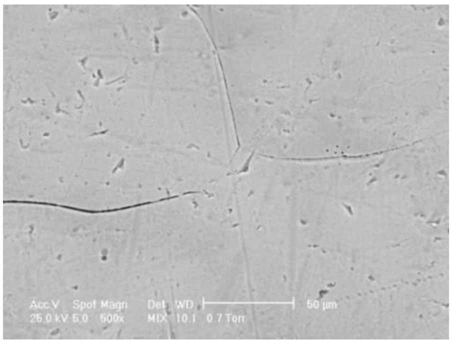


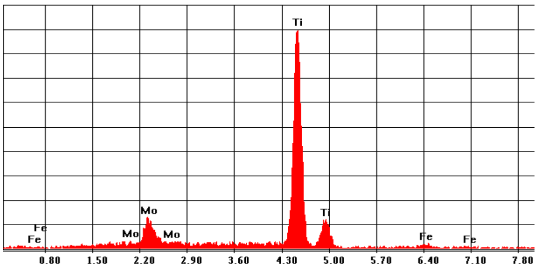
Ti15Mo5Fe x200

Fig. 2.2. Optical microstructures of the etched samples

The ESEM electronic microscope retains all of the characteristics of a standard SEM, but eliminates the inconveniences associated with the advanced vacuum that must be obtained around the sample, allowing us to analyse wet, oily, dirty, and nonconductive samples even in their natural state, without changing any of the actual parameters prior to sample preparation. In the electron microscopy laboratory, using this new type of microscope, images of secondary electrons and backscattered electrons can be obtained in advanced vacuum conditions and even in a water vapor environment, when analysing non-conductive or biological samples. The microscope is outfitted with an EDAX device, which allows for compositional, qualitative, and quantitative analyses, as well as the distribution of the elements in the composition of the analysed sample across its entire surface.

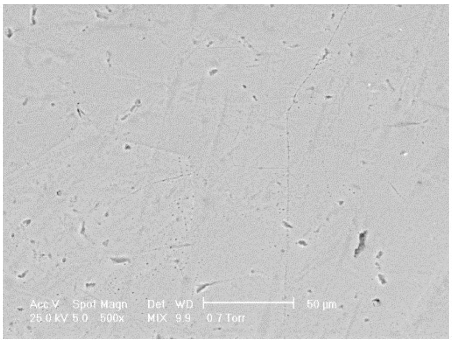
The SEM and EDS analyses performed with this microscope are shown below, as well as the tables with the mass and atomic concentrations of the Ti-Mo-Fe samples (Figs. 2.3, 2.4, 2.5, 2.6, 2.7).

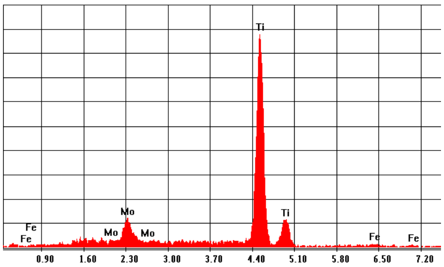




Element	Weight %	Atomic %
MoL	15.01	8.12
TiK	83.85	90.82
FeK	1.14	1.06
Total	100.000	100.000

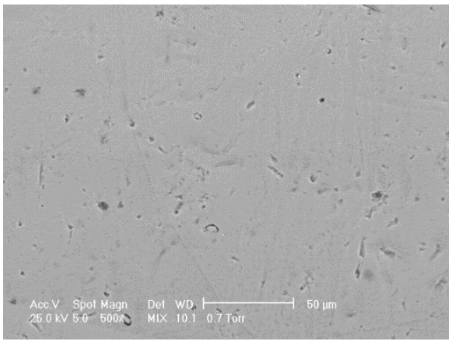
Fig. 2.3. SEM and EDS analysis of Ti15Mo1Fe alloy

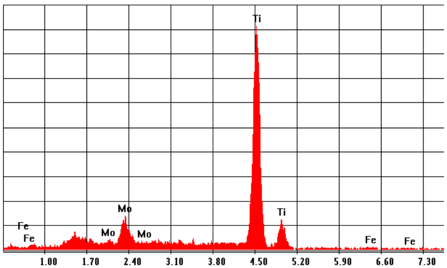




Element	Weight %	Atomic %
MoL	15.18	8.23
TiK	82.63	89.73
FeK	2.19	2.04
Total	100.000	100.000

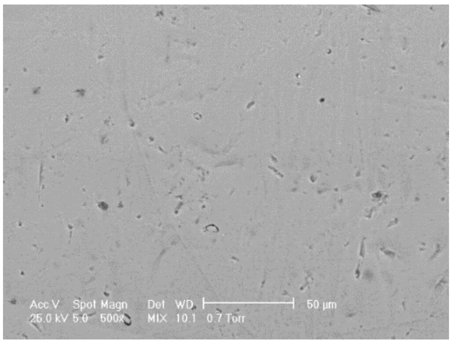
Fig.. 2.4. SEM and EDS analysis of Ti15Mo2Fe alloy

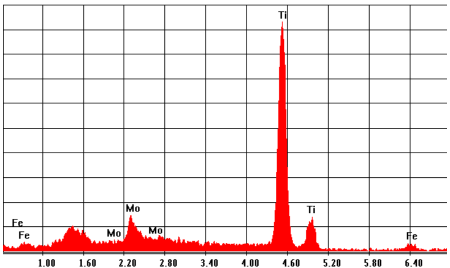




Element	Weight %	Atomic %
MoL	15.30	8.30
TiK	82.25	89.41
FeK	2.45	2.28
Total	100.000	100.000

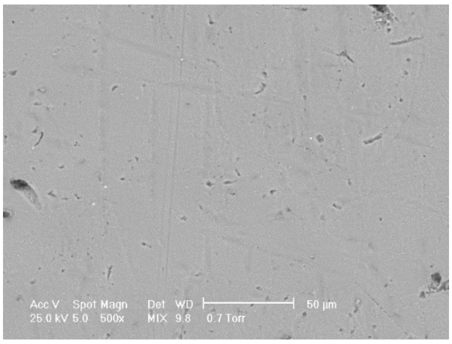
Fig.. 2.5. SEM and EDS analysis of Ti15Mo3Fe alloy

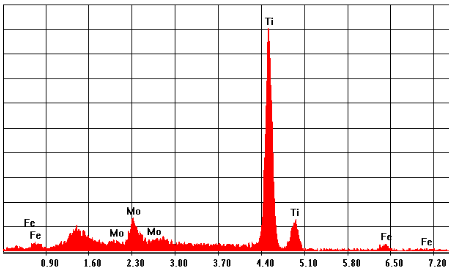




Element	Weight %	Atomic %
MoL	13.97	7.54
TiK	82.48	89.17
FeK	3.55	3.29
Total	100.000	100.000

Fig.. 2.6. SEM and EDS analysis of Ti15Mo4Fe alloy

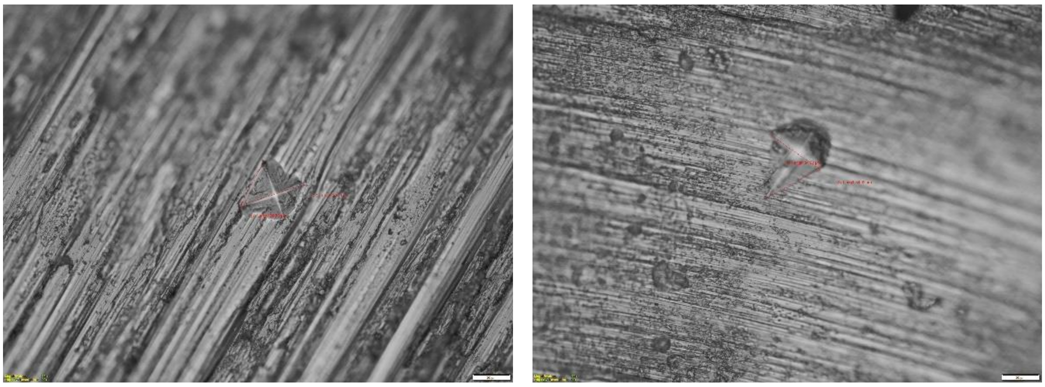




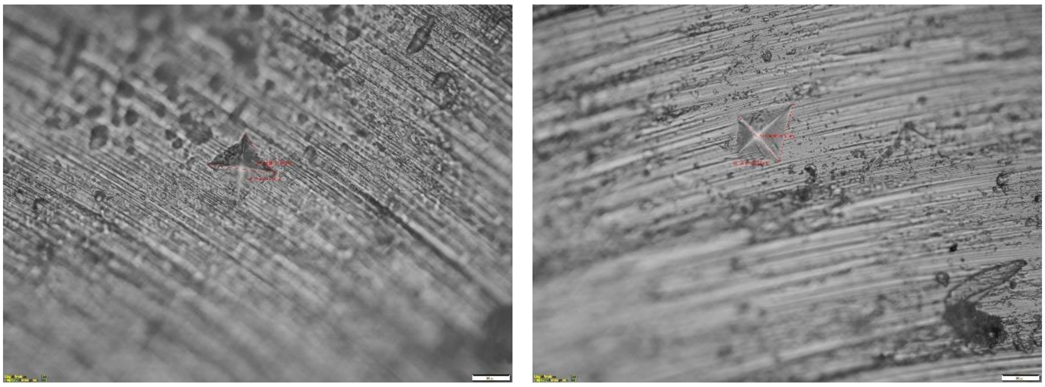
Element	Weight %	Atomic %
MoL	15.06	8.19
TiK	80.10	87.28
FeK	4.84	4.53
Total	100.000	100.000

Fig.. 2.7. SEM and EDS analysis of Ti15Mo5Fe alloy

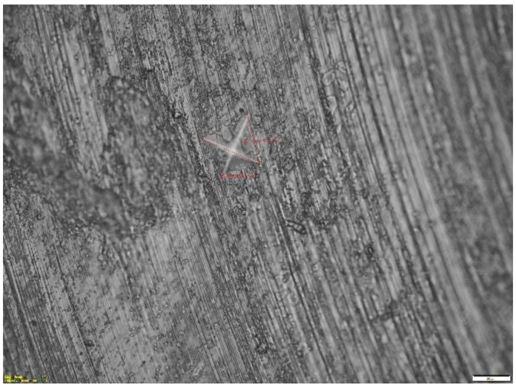
Vickers micro-hardness was measured with a Leco M-400-G device at a 300 g load for 15 seconds. Fig. 2.8 depicts the values for the indentation dimensions at x500 magnification.



Ti15Mo1Fe x500 Ti15Mo2Fe x500



Ti15Mo3Fe x500 Ti15Mo4Fe x500



Ti15Mo5Fe x500

Fig. 2.8. Optical microstructures of the samples after the microhardness test

Fig. 2.9 depicts the variation of the micro-hardness along with Fe concentration. Each sample was tested five times, and the average results are shown in the diagram as a black line, with each test represented by a bar. The average HV values (Table 2.3) decrease as more Fe is added, apart from the 5% Fe sample, which shows an increase in micro-hardness compared to the samples with a concentration of 3%Fe and 4% Fe.

Table 2.3

Values for the microhardness test and averages

Test No.	HV Microhardness				
	Ti15Mo1Fe	T15Mo2Fe	Ti15Mo3Fe	Ti15Mo4Fe	Ti15Mo5Fe
1	416.2	464.2	392.45	502.1	672.1
2	313.9	481.2	403.7	632	823.6
3	384.65	552.2	474.8	542.3	628.6
4	347.7	516.8	438.3	591.4	628.6
5	615.7	475.8	378.26	629.8	628.6
Average values	415.63	498.04	417.502	579.52	676.3

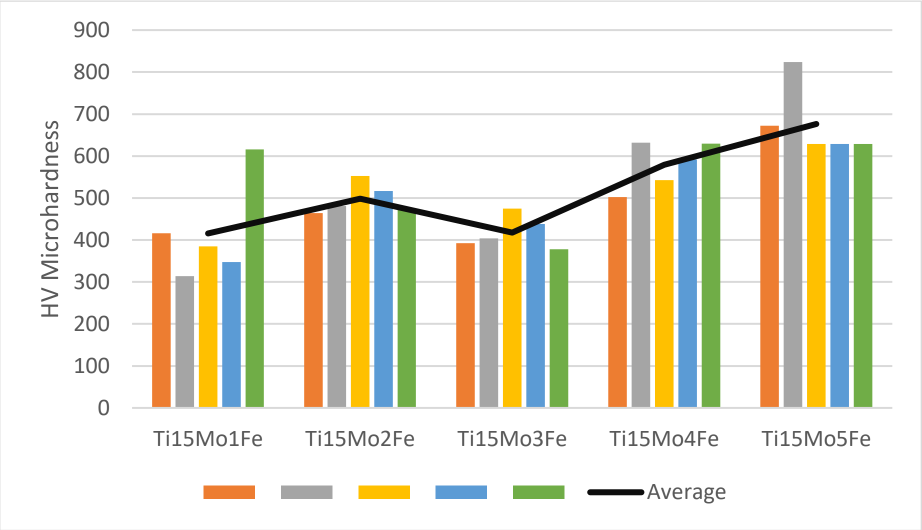


Fig. 2.9. Visual representation of the values for the microhardness test and averages

3. Conclusions

Special alloys, Ti-15Mo-(1%, 2%, 3%, 4%, 5% Fe), can be obtained by the electric arc method. The microstructures analysed are consistent with other structures present in the specialized literature.

This work has demonstrated the successful development of Fe alloyed Ti-Mo alloys which have promising properties such as low density and high strength.

Further studies are warranted to elucidate the long-term in vivo performance of these alloys. Fe alloyed Ti-Mo alloys offer promising possibilities for biomedical applications due to their improved mechanical properties and potential biological compatibility. Further studies are needed to better understand the effects of Fe addition on the microstructure, hardness, and mechanical behaviour in the Ti-Mo system.

Acknowledgement

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