THE EFFECT OF HEAT TREATMENT ON MICROSTRUCTURES, PROPERTIES AND OXIDE-SCALE THICKNESS OF TiAl-BASED INTERMETALLIC ALLOYS

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This study presents the results of the Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS) and X-ray diffraction (XRD) conducted on three of the Ti-52Al-5Cr; Ti-45Al-10Cr and Ti-45Al-15Cr alloys before and after a selected heat treatment process. X-ray diffraction and microscopy data prove that the as received state of Ti-52Al-5Cr alloy has DP microstructure consisting of alternate lamellae of α_2 (hcp) and γ (fcc) phases and the other grains, free of lamellar microstructure have γ phase. Heat treating of the alloy at 1100 °C for 24 hours with argon-gas flowing and a heating rate of 10 °C/minute followed by furnace cooling has led the finer grain sizes in the DP microstructure. The heat treatment route also produces the DP microstructure and lamellar structure in Ti-45Al-10Cr and Ti-45Al-15Cr alloy, respectively. Importantly, the selected heat treatment can improve the oxidation resistance of the alloys as indicated with the lowering of weight gain of heat treated samples alloys during the oxidation process inside a muffle furnace at 900 °C for 50 hours. Beside that the oxide-scale thickness of the oxidized heat treated alloys is less than that of the non-heat treated alloys. The improvement was postulated due to identified the lower volume fraction of α_2 and β phase (detrimental effect) and γ and Laves phase (beneficial effect) to oxidation resistance in the heat treated alloys than as-cast received alloys using XRD technique.

1. INTRODUCTION

New intermetallic TiAl-based alloys have great industrial interest due to their attractive properties for specific applications in aerospace, automotive, chemical process and power generation industries. Their high specific strength, good substantial mechanical properties at high temperatures and combining with low density (half that of steels) for weight savings, make these materials excellent candidates for high-temperature structural materials applications in the future. In these alloys the mechanical properties such as creep resistance, fatigue limit and specific strength are very sensitive functions of the microstructure [1].

As a function of the processing variables the microstructure can exhibit local changes of composition, morphology and distribution of the different phases, grains size and even texture. For this reason it is necessary to understand and quantify the influence of processing conditions of heat treatment on the microstructure and the corresponding to its properties. As structural materials applications which will be exposed at high temperature for long periods of time, it is very necessary to evaluate the microstructure and its oxidation resistance beyond to 700 °C. The variations in microstructures that can be controlled in the titanium aluminide alloys are numerous, but they exist in four broad groupings; that is near γ (NG), duplex (DP), near lamellar (NL), and fully lamellar (FL) microstructures. DP and FL microstructures are two typical microstructures which have been subjected to the most investigations. For this reason considerable effort has been devoted to developing processing techniques aimed at controlling the microstructure to optimize the specific properties with respect to the envisaged applications. A DP type, consisting of γ grains and lamellar colonies of alternating plates of (γ + α_2) phase, is one of commonly formed microstructures in cast conditions. Generally, a DP structure with fine grain sizes (d < 50 μm) is characterized by a better plasticity. Therefore, a properly designed heat treatment route should lead to the appropriate alloy phase composition and a high degree of grain refinement. Several heat-treatment processes which known in this time are: (i) thermomechanical processing, which enables to get FL microstructure during hot working; (ii) thermomechanical treatment, when a selected alloy chemistry containing boron as a grain-growth inhibitor is heat treated to form FL

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microstructure after hot working; and (iii) refinement, which enables both the grain size and lamellar spacing to be controlled, with or without grain-growth inhibitors through post-hot working heat treatment. All the processes take long time and more cost [1, 2, 3, 4].

In this study, a simply and effective of selected heat treatment process is offered as shown in Fig. 2. Thus, the aim of this study is to understand effects of the heat treatment process on the microstructure, and the influence on its properties including density, hardness and oxidation resistance of TiAl based alloys. The selected heat treatment route is depicted in Fig. 2. The three alloys used with nominal composition in this study are (A) Ti-52Al-5Cr, (B) Ti-45Al-10Cr and (C) Ti-45Al-15Cr alloy (as shown in Table-1). Characterization of the as-cast received and heat-treated alloys were performed using the Scanning Electron Microscope (SEM)/Energy Dispersive Spectroscopy (EDS) and X-Ray Diffractometer (XRD). Measurement of density and hardness was carried out on the alloys with a Micromeritics Acupy, Gas Pycnometer 1330 and Vickers's hardness tester, respectively.

2. EXPERIMENTAL PROCEDURE

The comparative study was carried out in three alloys, namely A, B, and C of respective nominal compositions in atomic percentage as shown in Table-1. The alloys were prepared by the specially-designed and locally-made arc-melting furnace with a non consumable electrode under argon atmosphere in a water cooled copper hearth. The raw materials used were Ti and Al high purity pellet powders (Fig. 1a). A previous and normally casting procedure was established in order to obtain as-cast buttons (Fig. 1c) alloy free from macro-segregation, with ~ 6 gram of weight, 20 mm in diameter and 10 mm in thick. The buttons were remelted 5 times to ensure chemical homogeneity. The figure of the arc-melting furnace can be seen in Fig. 1b. Cubic specimens were cut from ingot using diamond cutting tool machining. They were heat treated in tube furnace at 1100 °C with heating rate at 10 °C/min for 24 hours and furnace cooled. The selected heat treatment route is schematically illustrated in Fig. 2. The variatious hardness and density as a function of heat-treated and non heat-treated was evaluated by Vickers's hardness tester and a Micromeritics Acupy, Gas Pycnometer 1330.

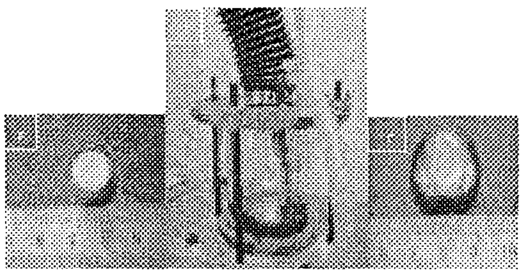


Fig. 1. (a) a pellet of metals powder; (b) a cell of arc-melting furnace; and (c) a button of alloy [5].

Table I. Some selected nominal compositional variations of alloys in at.%.

Name of alloy	*Various Compositions*	*Atomic %*		
		Ti	*Al*	*Cr*
A	Ti – 52Al-5Cr	43	52	5
B	Ti – 45Al-10Cr	45	45	10
C	Ti – 45Al-15Cr	40	45	15

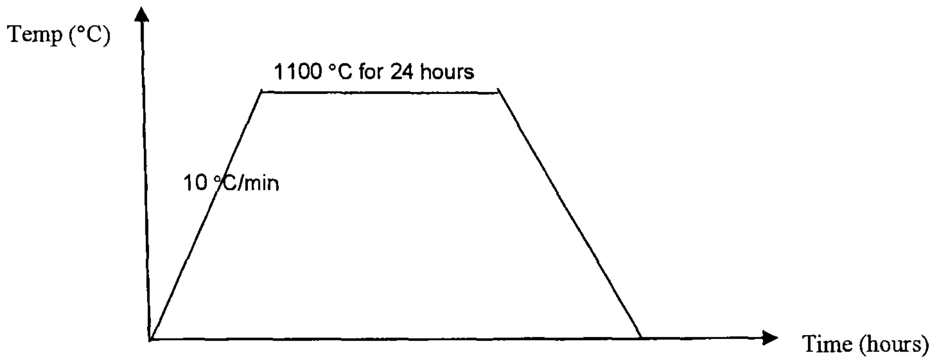


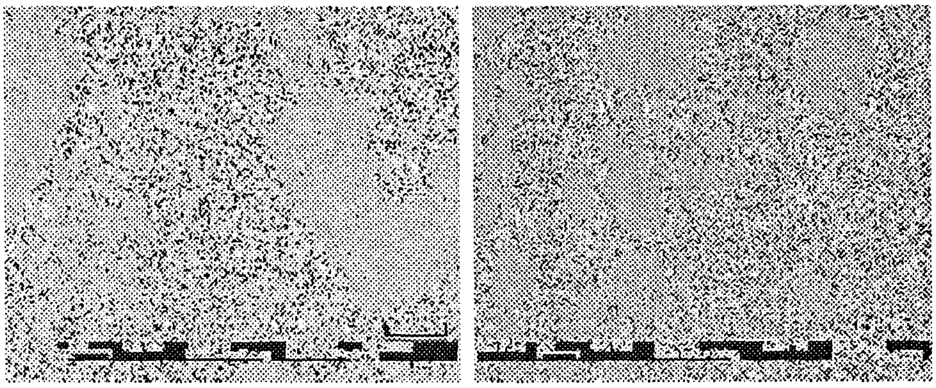
Fig. 2. Schematically temperature-time path used of the heat treatment process.

For microstructural studies, metallographic samples were prepared in a standard fashion to reveal the structure. The specimens were then ground to a mirror like surface with SiC papers up to 2000 followed by 0.1 and 0.05 μm alumina powders. Finally, the ground surface of specimens was etched in a modified Kroll's reagent of 10 vol. % HF, 4 vol. % HNO_3 and 86 vol. % H_2O. The microstructures were studied by a LEO SUPRA 50VP electron microscope using SEM/EDS. In addition, X-ray Diffraction (XRD) technique with Siemens Diffractometer D5000 is also used to confirm the results of SEM/EDS. Oxidation processes have been carried out on the other cubic specimens in opened air under isothermal condition of 900°C for 50 hours inside a muffle furnace. The weight gain of the specimens during the oxidation process was determined. And cross-sectional microstructures of the samples have been characterized using SEM/EDS technique.

3. RESULTS AND DISCUSSION

3.1 Microstructure Studies

The as-cast microstructures in the three alloys before and after heat treatment are presented in Fig. 3, 4, and 5. Fig. 3a shows the microstructures of the as-cast sample of Ti-52Al-5Cr alloy before heat treatment process. It shows a duplex structure (DP). Generally, a DP type microstructure is characterized by a better plasticity and adequate tensile ductility, but it is inferior to a lamellar microstructure with respect to crack resistance, fatigue strength and high-temperature creep resistance [2]. Many grains in the microstructure exhibited a columnar morphology with a colony width of ~ 40 μm and length of ~ 70 μm. Within the colony grains, the microstructure consisted of lamellae of α_2 (hcp) and γ (fcc) phases. The other grains, free of lamellar microstructure have γ phases with size of ~ 80 μm. With aid of EDS, microanalytic study has shown that the chemical composition of the grains i.e.: 49.84 at.% Al, 42.50 at.% Ti and 7.65 at.% Cr for lamellae α_2/γ and for γ phase of 54.60 at.% Al, 42.39 at.% Ti and 3.01 at. % Cr. The Al content in the regions consisting of lamellae structure is greater than its Ti content. Thus, the predominating component in the lamellar structure is the γ-TiAl phase poor in Ti, according to equilibrium system, is composed of a mixture of alternating ($\alpha_2 + \gamma$) plates [6]. Meanwhile, in the regions of free lamellar microstructure have an increased Al content (above 50 at. %), which shows that it is the γ-TiAl phase. It was confirmed using XRD results as in Figure 6. Clearly, the diffractogram patterns show that γ-TiAl as predominating present phase or primary phase in the alloy.



(a) (b)

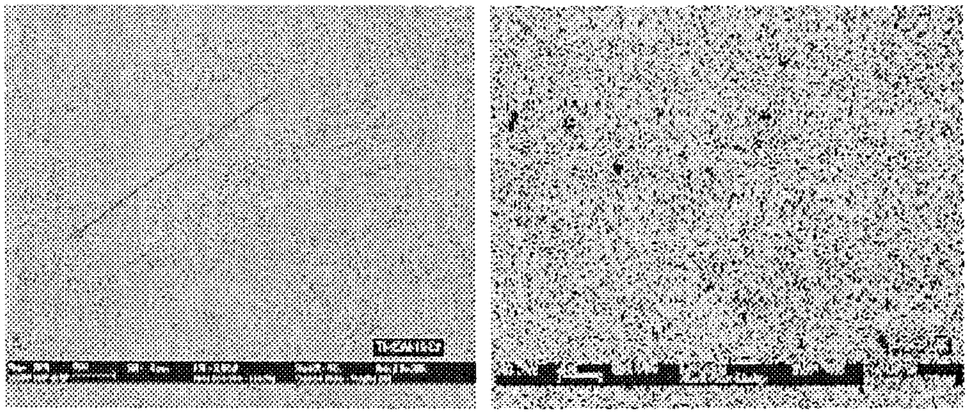
Fig. 3. Typical lamellar grains observed of the Ti-52Al-5Cr by SEM micrograph with magnification of 1000x (a) before heat treatment; and (b) after heat treatment

On the other hand, the microstructure of heat treated Ti-52Al-5Cr alloy is depicted in Figure 3b. It shows that the microstructure of heat treated is similar with non heat-treated alloy, i.e. duplex type. It also includes regions consisting of lamellae α_2/γ phase and γ phase region. But the grain size of the heat treated microstructure is finer than non-heat treated one and it's preferred. The grain size, defined by the length of the lamellae is ~ 30 μm and width is ~ 20 μm. Meanwhile for γ-TiAl phase (free lamellae structure) the grain size of ~ 25 μm. So the heat treatment process route works successfully for grains refinement of microstructure type of duplex structure. The grain size refining was developed only in one process and resulted 50 % of finer grains. In addition, the uniform of grains is observed on heat treated microstructure of Ti-52Al-5Cr alloy. The diffractogram patterns of XRD on heat treated alloy shows the lower of α_2 phase. As well known, the lower of volume amounts (about 5 – 20%) of α_2 phase and higher of γ phase is expected to obtain the best properties of an alloy. Brady reported that the presence phases of γ-TiAl, $TiAl_2$ and Laves $Ti(Al,Cr)_2$ phase in the alloys can improve their oxidation resistance during exposure at high temperatures. On the contrary, the existences of α_2-Ti_3Al and β phases give the detrimental effect for their oxidation resistance because they have high oxygen permeability. In that condition, Ti element and oxygen widely diffusion to form oxide scales during oxidation which can increase its mass gain, so the oxidation resistance of the alloys will decrease.

Fig. 4a and 5a shows the as-cast microstructures of Ti-45Al-10Cr (B) and Ti-45Al-15Cr (C) alloy respectively. The both of microstructures are different but the present phases of the alloys are similar. It consists of β phase as the primary phase and α_2 phase as the second phase. Dendrites typical of microstructure is observed on B alloy whereas the direction of solidification as shown in the sign in Fig. 4a. It presents the long bright gray dendrites and no interdendritic phase observed. The dendrites morphology showed six-fold symmetry or hexagonal dendrites morphology [7]. The dendrites has a chemical composition of ~ 11.27 at.% Al, 87 at.% Ti and 1.44 at.% Cr and that it is a α_2 phase. Meanwhile for regions free dendrites as a matrix in the B alloy, the chemical composition is ~ 36.06 at.% Al, 52.08 at.% Ti and 11.86 at.% Cr which identified as β phase. On the other hand, chemical composition on the C alloy, is 48.85 at.% Al; 42.14 at.% Ti and 9.01 at.% Cr for present phase of β. Meanwhile for present phase of α_2, is 23.84 at.% Al; 73.74 at.% Ti and 2.42 at.% Cr. The existence of β phase as primary phase in as-cast received of the alloy give the detrimental effect for degree of its oxidation resistance.

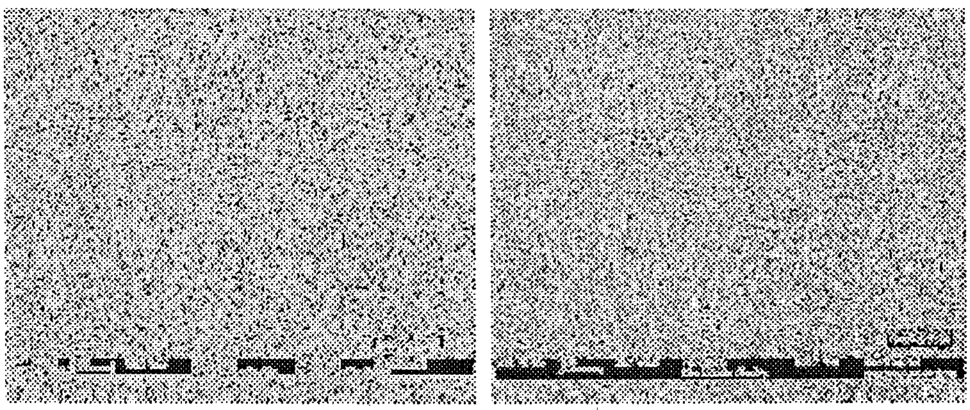
However, in heat-treated B alloy, the present phase of β reveals in a small amount and not as the primary phase. That condition is better and expected to improve the oxidation resistance. Both of heat treated microstructures of alloys reveal amount regions of lamellar structure. Typical microstructure of the alloys are duplex, same with the previous Ti-52Al-5Cr alloys but not with clearly grain boundaries. It consists of lamellae α_2/γ phase and γ-phase. In some areas, dendrites grains of α_2 can be still observed. Their grain size of the B alloy is ~ 25 μm in length and ~ 20 μm in width for lamellae structure and the size of γ phase is ~ 25 μm. With aid of EDS, the chemical compositional of present phase can be identified. They are lamellae (α_2/γ) structures with ranging compositions i.e.: 2.79 at.% C, 40.90 at.% Al; 49.02 at.% Ti and 7.29 at.% Cr for γ phase and 15.76 at.% C; 27.29 at.% Al, and 56.94 at.% Ti for α_2 phase. The phase is on typical of dendrites but the grain size is not clear in case of C alloy.

Usually, the methods mainly used for refinement are: supertransus processing (hot extrusion and forging), thermo mechanical treatment (forging plus heat treatment), solidification with grain refiner, and rheocasting. The all methods involve mechanical treatment at high temperatures and even in vacuum, and therefore induce significant difficulties in experimentation [4]. In this study, the achievements are successfully obtained only by selected heat treatment which may be cost-effective in a commercial sense.



(a) (b)

Fig. 4. Typical grains observed of the Ti-45Al-10Cr by SEM micrograph in the secondary electron mode with Mag. 500x (a) before heat treatment; and (b) after heat treatment.



(a) (b)

Fig. 5. Typical grains observed of the Ti-45Al-15Cr by SEM micrograph in the secondary electron mode with Mag. 1000x (a) before heat treatment (b) after heat treatment

3.2 XRD Results

The quantitative phase analysis was performed in order to identify the exact set of phases before and after the heat treatment route process. The examples of results of fitting procedure for experimental X-ray diffraction patterns using PC-APD for Windows Ver. 4.0g software manufactured by Philips Electronics N.V. 1999 are shown in Fig. 6, 7 and 8. All the diffraction patterns indicate the present phases of γ, α_2, β and it's in agreement with literature reports and previous SEM/EDS results [8, 9].

Fig. 6 shows the X-ray diffraction patterns of Ti-52Al-5Cr alloy. It indicates that the alloy is in the two phase state: γ phase ($L1_0$) and α_2 phase (($D0_{19}$). It is agreement with the observed of SEM that shows the duplex microstructure consisting of equiaxed grains with the γ single phase and the lamellar colonies of ($\gamma + \alpha_2$) phase. The important point from this characterization that is the heat treatment process significantly can decrease the volume fraction of α_2-phase in the alloy.

Fig. 7 shows the patterns of Ti-45Al-10Cr alloy. Its diffraction patterns show vividly the presence of the third phase, bcc ordered β(B2) phase namely, due to the presence of Cr alloying element in the composition. It's similar with Ti-45Al-15Cr alloy as can be shown in Fig. 8. In both of figures, present phases identified are γ, α_2 and β.

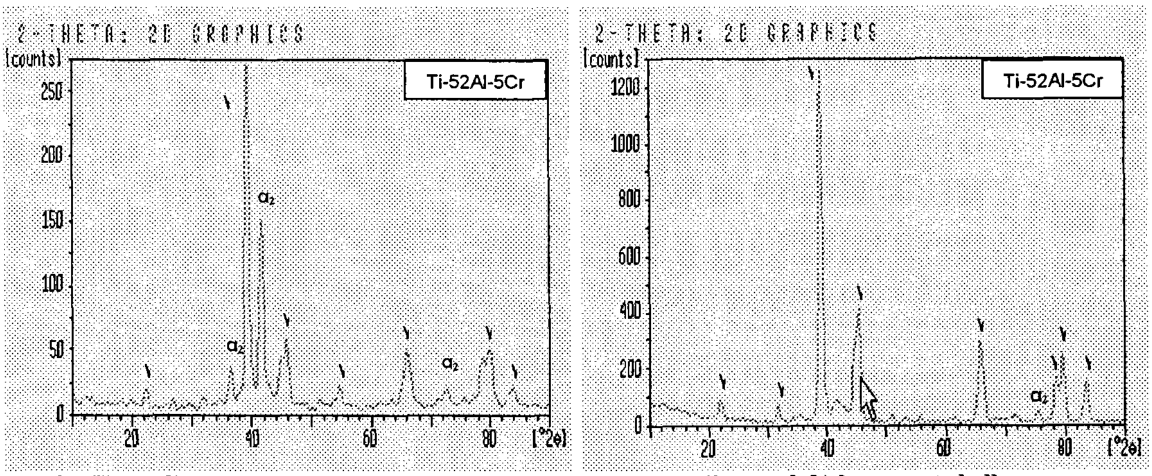


Fig. 6. X-ray diffraction patterns of Ti-52Al-5Cr alloy (a) as-cast alloy; and (b) heat-treated alloy.

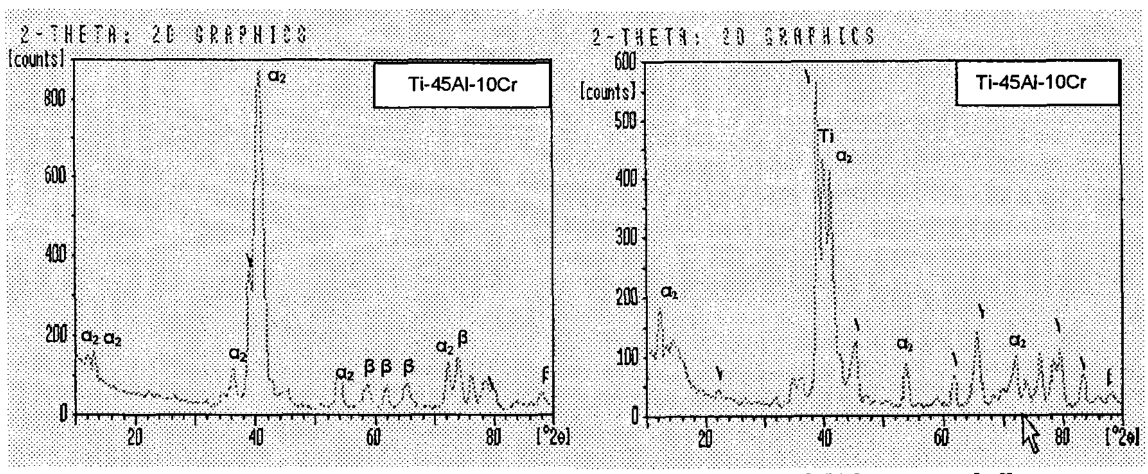


Fig. 7. X-ray diffraction patterns of Ti-45Al-10Cr alloy (a) as-cast alloy; and (b) heat-treated alloy.

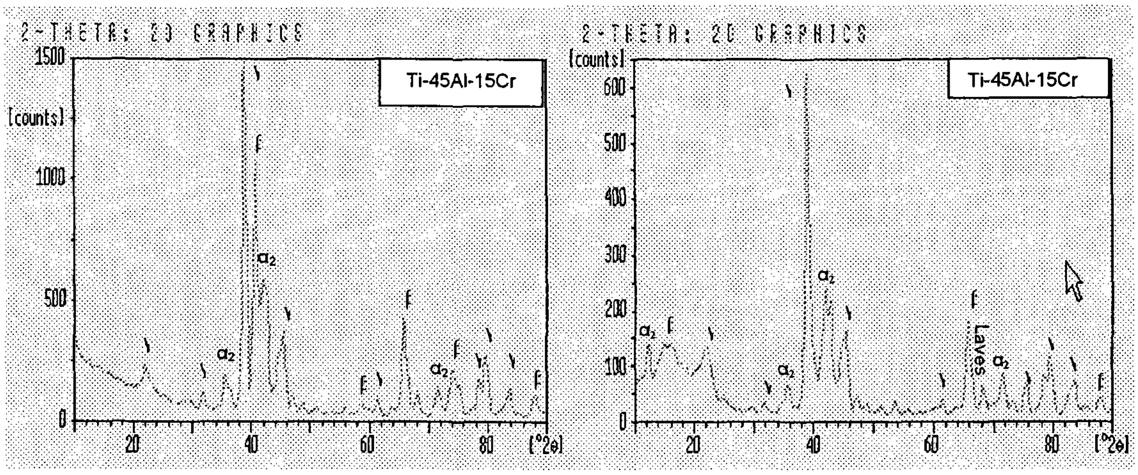


Fig. 8. X-ray diffraction patterns of Ti-45Al-15Cr alloy (a) as-cast alloy (b) heat-treated alloy.

3.3 *Density and Hardness Analysis*

Average density (specific weight) was done using Micromeritics AccuPyc 1330, Gas Pycnometer and Hardness test was carried out using Vickers Hardness Tester with loads of 10 kgf. The value of both of tests can be seen from the Table-2. The lower specific weight is more expected in the alloys. Specific weight increases with the decrease of aluminum content which raising α_2-Ti_3Al and TiAl phase [10]. It occurs due to density of Ti element is 4.5 gr/cm^3 greater than Al element is 2.7 gr/cm^3. The specific weight also increases with increasing of Cr content. It has very reason due to density of Cr element is 7.2 gr/cm^3. In this study, the highest value of density occurs on Ti-45Al-15Cr at 4.49 gr/cm^3, meanwhile the lowest is achieved on Ti-52Al-5Cr at 4.03 gr/cm^3. It can be seen that the selected heat treatment process which offered in this study can improve the specific weight of the alloys whereas their density of heat treated alloy is lower than as-cast received alloys. Its low density, which is less than half of that of superalloys, is an important attribute for gas turbine engine applications. Lightweight materials increase the engine performance as measured by thrust-to-weight ratio. The benefit can be more than pound for pound (weight) savings, since the use of a lightweight rotating part frequently reduces the dimensions of the stress-supporting structures. Additionally, the high aluminum content of this compound increases the resistance of oxidation and burning, two concerns in the use of titanium-based materials.

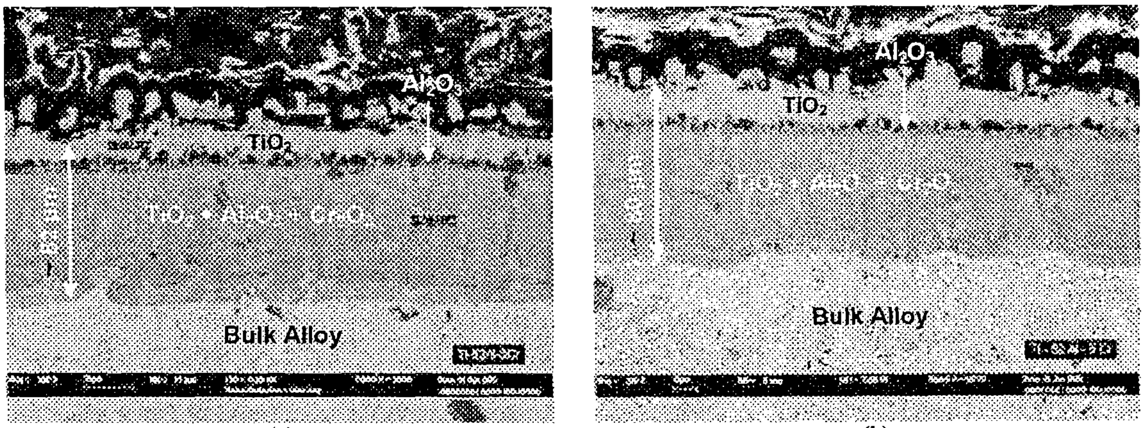
Table II. Average density and hardness value of the alloys

Name of Alloys	Density (gram/cm^3)		Hardness Vickers (VHN)		Tensile Strength (MPa)	
	Before HT	After HT	Before HT	After HT	Before HT	After HT
Ti-52Al-5Cr	4.03	4.03	524	560	5138.868	5491.92
Ti-45Al-10Cr	4.30	4.28	640	645	6276.48	6325.515
Ti-45Al-15Cr	4.49	4.40	674	673	6609.918	6619.725

Table-2 shows the Vickers average hardness increases with decreasing of Al content of TiAl-based alloys, generally. The properties of TiAl-based alloys are strongly composition and microstructure-dependent. Alloying elements dominate the strengths of alloys for given microstructures. Al is the most influential element to the alloy strength and it acts through changing the volume fraction of the α_2-phase, which is the hard phase in ($\alpha_2 + \gamma$) two-phase alloys [10]. With a decrease in Al concentration the α_2 volume fraction in increased and so is the strength. Other elements like Cr seem to have as large an effect on alloy strength solid solution up to a certain temperature. As shown in Table-2, for the alloys increasing amount of Cr in the TiAl-based alloys can increase the Vickers average hardness, significantly. The highest value reached at VHN = 674 of Ti-45Al-15Cr and the lowest occurred at VHN = 524 of Ti-52Al-5Cr alloy. And same with the specific weight, the heat treatment process in this study can improve the specific strength of the alloys. In addition, to convert VHN to MPa multiply by 9.807 [13].

3.4 *Oxidation of Alloys*

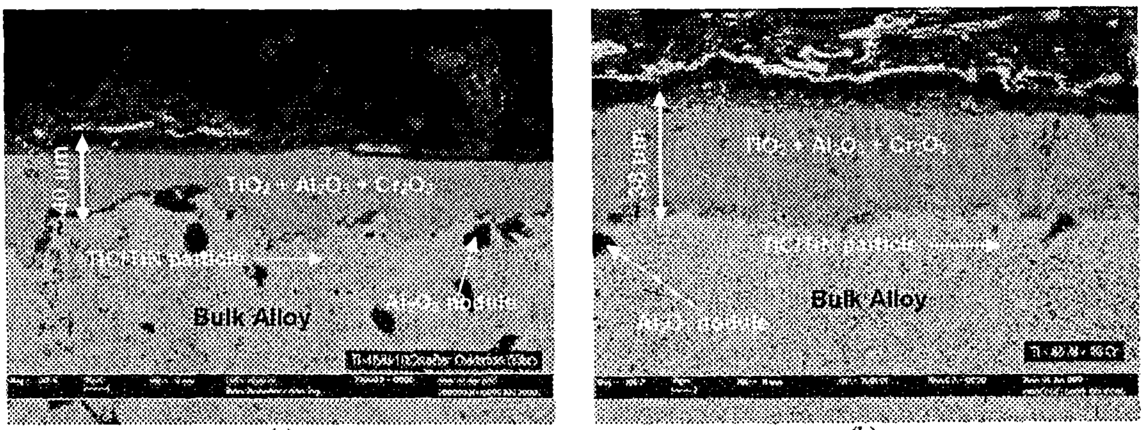
Scale cross-sections of Ti-52Al-5Cr alloy after isothermal oxidation processes for 50 hours at 900 °C in opened air inside a muffle furnace, are shown in Fig. 9. Fig. 9a presents the scales of non heat-treated alloy while Fig. 9b shows the heat-treated alloy. It's found that the microstructures of bulk alloys remain unchanged i.e.: lamellar structure. The whole scale thickness was ~ 76 μm for non heat-treated and ~ 74 μm for heat-treated alloy. It shows that both of the alloys have the nearly similar oxidation resistance. The weight gain after the oxidation processes exhibits the postulate. During processes of oxidation, Ti-52Al-5Cr alloy exhibit decreasing weight gain of 0.95% and 0.91% for non heat-treated and heat-treated alloy, respectively.



(a) (b)

Fig. 9. Cross-sectional microstructures after oxidation in air at 900 °C for 50 hours of Ti-52Al-5Cr alloy with magnification 500x (a) before heat treatment; and (b) after heat treatment.

Three types of oxide scales have been produced as the results of oxidation processes for both of the alloys: TiO_2, Al_2O_3 and (TiO_2+Al_2O_3). The thickness of the outer scale, TiO_2 is ~ 18 μm and ~ 16 μm for non heat-treated and heat-treated alloy, respectively. On the other hand, the inner intermixed layer of (TiO_2 + Al_2O_3) is ~ 58 μm for both of the oxidized alloys. The both of scales, outer and inner layer, were separated by nearly continuous layer of Al_2O_3 with thickness of ~ 6 μm.

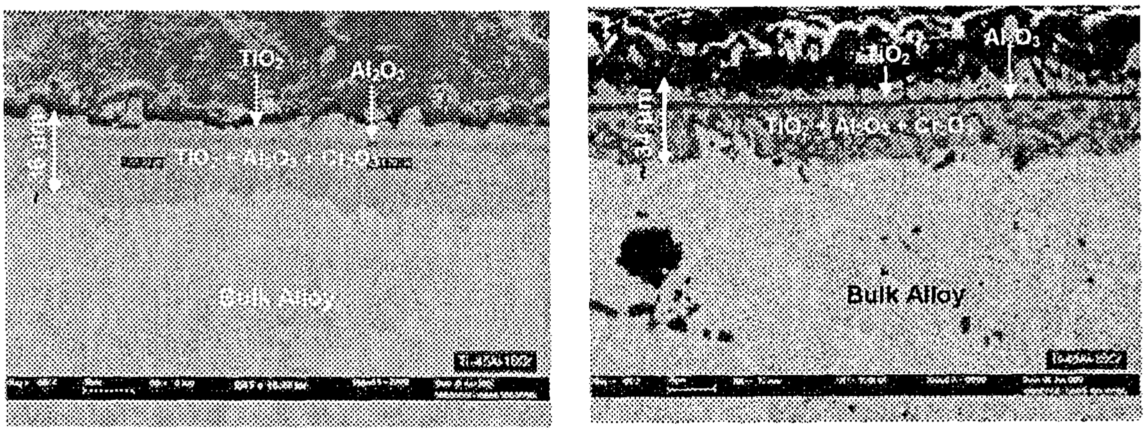


(a) (b)

Fig. 10. Cross-sectional microstructures after oxidation in air at 900 °C for 50 hours of Ti-45Al-10Cr alloy (a) before heat treatment (with Mg. 250x); and (b) after heat treatment (with Mg. 500x).

Fig. 10 presents the oxidation results on Ti-45Al-10Cr alloy. The morphologies scales of the oxidized alloys are very similar. The cross-sectional microstructures of the alloy is showing only one mixed scale of (TiO_2 + Al_2O_3 + Cr_2O_3) exists on the surface of the alloy. The thickness of the scale is ~ 40 µm and ~ 38 µm for non heat-treated and heat-treated alloy, respectively. During processes of oxidation, Ti-A45l-10Cr alloys exhibit decreasing weight gain of 1.43% and 1.40% for non heat-treated and heat-treated alloy, respectively. In accordance with the weight gains, the oxidation resistance of Ti-45Al-10Cr alloy is poorer than the previous of Ti-52Al-5Cr alloy due to the inexistence of alumina scale in Ti-45Al-10Cr.

Within the bulk of Ti-4Al-10Cr alloy, particles of TiN/TiC can be identified. The formation of TiN/TiC phase which interrupts the establishment and formation of a continuous alumina scale is considered to be detrimental due to the inability of the alloy to form a protective alumina scale. The existence of alumina (Al_2O_3) nodules inside the bulk alloy also shows the insufficient of oxidation resistance of Ti-45Al-10Cr alloy compared to Ti-52Al-5cr alloy. It means internal oxidation (unexpected condition) have been occurred in the alloy. Thus, the existence of the nearly or full continuous and protective alumina thin scale is needed to protect the bulk of alloys [8, 9].



(a) (b)

Fig. 11. Cross-sectional microstructures after oxidation in air at 900 °C for 50 hours of Ti-45Al-15Cr alloy with Magnification 500 (a) before heat treatment (b) after heat treatment.

On the other hand, similar situation with Ti-52Al-5Cr alloy occurred for Ti-45Al-15Cr alloy having layers of TiO_2 scale on the surface layer, followed by Al_2O_3 (alumina) scale between the outer layer and the inner layer of mixed (TiO_2 + Al_2O_3 + Cr_2O_3) scale (as shown in Fig. 11). The whole scale thickness was ~ 36 µm for non heat-treated and ~ 34 µm for heat-treated alloy.

The surface layer of the oxide scales is made of ~ 7.0 µm thick of TiO_2 scale sitting on top of the alumina layer with thickness of ~ 1.8 µm. The (TiO_2 + Al_2O_3 + Cr_2O_3) mixed scale of ~ 27.5 µm is also present beneath the previously mentioned outer scale. The situation occurred for non heat-treated of Ti-45Al-15Cr alloy (Fig. 11a). While, cross-sectional microstructures on heat-treated of Ti-45Al-15Cr alloy is given in Fig. 11b consisting of ~ 5.7 µm thick of TiO_2 scale, ~ 1.8 µm of Al_2O_3 scale and 25.8 µm. During processes of oxidation, Ti-45Al-15Cr alloys exhibit decreasing weight gain of 0.63% and 0.62% for non heat-treated and heat-treated alloy, respectively.

4. CONCLUSION

Characterization with SEM/EDS and XRD show that the as-cast received of Ti-52Al-5Cr alloy has DP microstructure consisting with lamellae of (α_2 + γ) dual phase and single phase of γ. In Ti-45Al-10Cr and Ti-45Al-15Cr, the initial microstructures were dendrites type and grains microstructure, respectively. Heat treating of the alloys at 1100 °C for 24 hours with argon-gas flowing and a heating rate of 10 °C/minute followed by furnace cooling has led the changing of the microstructure.

The grain size of DP structure in heat treated of Ti-52Al-5Cr alloy is finer than as-cast received alloy. On the other hand, the DP microstructure and lamellar structure in Ti-45Al-10Cr and Ti-45Al-15Cr alloys respectively were produced after heating on the both of alloys. The heat treatment process which offered in this study can improve the specific weight of the alloys whereas their density of heat treated alloy is lower than as-cast received alloys. The highest value of density occurs on as-cast received and heat treated of Ti-45Al-15Cr alloy at 4.49 and 4.40 gr/cm^3 meanwhile the lowest is achieved on both of Ti-52Al-5Cr alloys at 4.03 gr/cm^3. The highest hardness value was reached at VHN = 674 of Ti-45Al-15Cr and the lowest occurred at VHN = 524 of Ti-52Al-5Cr alloy. Similar with the specific weight, the selected heat treatment process in this study can improve the strength of the alloys.

Importantly, the selected heat treatment route can enhance the oxidation resistance of the alloys as indicated with the lowering of mass gain of the heat treated samples alloys during the oxidation process inside a muffle furnace at 900 °C for 50 hours. The oxide-scale thickness of the oxidized heat treated alloys is less than that of the non-heat treated alloys. The improvement was postulated due to identified the lower volume fraction of α_2 and β phase which detrimental effect and higher of γ phase which beneficial effect to oxidation resistance in the heat treated alloys than as-cast received alloys using XRD technique.

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