E3 Journal of Scientific Research. 2(2). pp. 026-034, August, 2014
Available online http://www.e3journals.org
ISSN: 2276-9897 © E3 Journals 2014

Full Length Research Paper

Fabrication and characterization of Al-based in situ composites reinforced by Al_3V intermetallic compounds

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Accepted 18 April, 2014

Al-V insitu composite reinforced by submicron size Al_3V intermetallic compound has been obtained using new technique. The insitu Al_3V intermetallic compound was homogeneously embedded in the Al matrix. It is produced by mixing vanadium pentoxide with powdered aluminium for 6 hr in a cylindrical polyethylene bottle using a ZrO_2 ball. The mixture was added to molten aluminium at different temperature and different Al powdered/V_2O_5 Wt. ratio. The produced composites were examined using XRD, SEM, EDS, DTA, light microscopy and hardness testing. The results indicated that, firstly the powder aluminium reduced the V_2O_5 to librate vanadium ions, the later reacted with molten aluminium to form the required composite. The recovery of vanadium in the prepared composite is reached up to 8% and the maximium efficiency of the V recovery up to 80% has been achieved. Also, it has proved certainly by introducing many evidence using XRD, SEM, EDS, DTA and X ray mapping that, this compound is Al_3V intermetallic compound. According to DTA analysis, the reduction reaction is exthothermic reaction at temperature more than 600°C and the bath temperature uncontrolled reached up to 1000°C. The presence of Al_3V compound in the aluminium matrix enhanced and improved the hardness of the prepared composite.

Keywords: Al-V alloys, Vanadium pentoxide, Al_3V intermetallic compound

INTRODUCTION

Al-V master alloys, for use in Ti-Al-V alloys for aerospace and other applications. Aluminium-transition metal alloys exhibit superior mechanical properties such as vanadium and titanium, these alloys have high thermal stability and corrosion resistance. Accordingly, these alloys can be applied for wide range of aerospace applications, missiles and air frame structure (Woo and Lee, 2007). Al-V alloys is being produced to meet the highest quality and reliability requirements of the aerospace industry. It is an alloy that can strengthen titanium which is used in critical parts of aircraft as a result, the alloy must meet rigid quality standards (Omran, 2007). Al-V master alloys is also added to titanium for producing Titanium-Aluminum-Vanadium alloy (Ti-6Al-4V) which has been used as a biomaterials becouse of its biocompatibility and good mechanicl properties (Stolecki et al., 1987).

On the other hand, Metal matrix composites (MMCs) reinforced with ceramic particles, whisker, or fiber have commonly been used in car manufacturing, railway train and space aircraft industries because of their high specific strength, superior wear resistance and other excellent mechanical properties (Woong-Seong and Muddle, 1997; Woo et al., 2010). The intermetallic compounds of Al-Zr, Al-Ta, Al-Nb, Al-W and Al-Mo in Al matrix could reduce the differences of the thermal expansion coefficient of the composite. These intermetallic compounds have high hardness and Young's modulus. In particular, the reaction between V_2O_5 and Al during mechanical milling has been studied (Yang and McCormick, 1994). This is because V_2O_5 and Al reacted together and were transformed into new phases at a high temperature. Phase analysis based on the differential thermal analysis (DTA) and X-ray diffraction (XRD) analysis.

The raw materials of vanadium include vanadium pentoxide, ferrophosphorus slag, petroleum residues, spent catalysts, utility ash, and vanadium bearing iron slag (Omran, 2006; Woo et al., 2010; Kuwabara et al., 2000). Generally, transition-metal trialuminide intermetallic could provide the kind of reinforcement for

Table 1. the experimants input, output and processing temperatere

Wt. of V_2O_5	Wt. of Al powder, gm	Wt. of molten Al, gm	Al powder/ V_2o_5	Temperatere, °C	V in composite, Wt. %
30	0	165	0	800	0
30	7.5	172.5	0.25	800	3.5
30	15	187.5	0.5	800	8.3
30	22.5	195	0.75	800	8.8
30	15	187.5	0.5	700	7.3
30	15	187.5	0.5	750	7.8
30	15	187.5	0.5	850	8.0

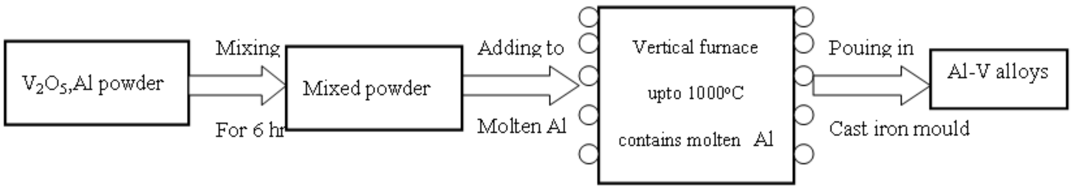

Figure 1. Schematic diagram for experimental setup

light metal matrices. These intermetallics having low densities and high elastic mouduli, are good candidates for in-situ reinforcement of light metal matrices based on aluminium alloys (Volkov and Gyrdasova, 2000; Varin, 2002).

The present work aims to study a novel technique for preparation of Al-V insitu composite reinforcd by Al_3V intermatallic compound by Mixing the V_2O_5 and Al using ball mill. The mixture is reacted with molten aluminium. An attempt to investigate the different factors affecting the preparation of composite such as temperature and Al powdered/V_2O_5 Wt. ratio. Because Al_3V intermetallic compound is an unknown compound in open literatures, so the other aim in this study is to prove certainly by introducing many evidence using XRD, SEM, EDS, DTA and X ray mapping that, this compound is Al_3V intermetallic compound.

Experimental

The materials used in this work were: Pure Al (99.9 %) powder (average particle size 59 µm); V_2O_5 (99.5 %) powder (average particle size 59 µm); and bulks alminium. A 30 g V_2O_5 was mixed with Al powder at different weights of Al powder, 7.5, 15, 22.5 g. The powders were uniformly mixed in cylindrical polyethylene bottle using a ZrO_2 ball with ball-to-powder ratio of 6:1 using horizontal mixing machine (Mechanical mixer, ABB ACS100), 150 rpm speed mixing time 6 hours; then the mixure was tested using DTA up to 900°C. The experiments were preformed in a vertical muffle furnace with temperature controller, and it contained silicon carbide crucible; the experimental setup is shown in Figure 1. A 99.7% purity of aluminium was melted in the silicon carbide cricible at elevated temperature varied from 700-850 °C; then a calculated amount of the mixed powder that was previously prepared was added to the molten aluminium with manual stirring maintained for 5 minutes followed by scamming the formed slag before pouring into suitable cast iron moulds to carry out the required tests. The chemical analysis of the produced alloys were carried out using Inductively Coupled plasma (ICP), Australian model. microscopic examinations were performed using light microscopy provide with image analyzer; and scanning electron microscopy SEM(JSM-6400 SEM) operated at 15 to 25 kV, equipped with an energy dispersive X-ray spectroscopy (EDS) was used to analyze the concentration of the constituent elements. X-ray diffraction analysis (XRD), Germany model using Cu radiation set at, step size 0.02° and step time 0.1. The factors affecting the preparation of the insitu Al-Al_3V composite are indicated in Table 1; these factors are: Bath temperature; Al/V_2O_5 weight ratio (R)

Results and Discussions

The experiments were carried out by adding a mixture containing vanadium pentoxide (source of V), and powdered (Al reduction agent) to molten aluminium. The factors affecting the recovery of vanadium in the produced Al-Al_3V composite were studied, these factors are: Bath temperature; Al/V_2O_5 weight ratio (R).

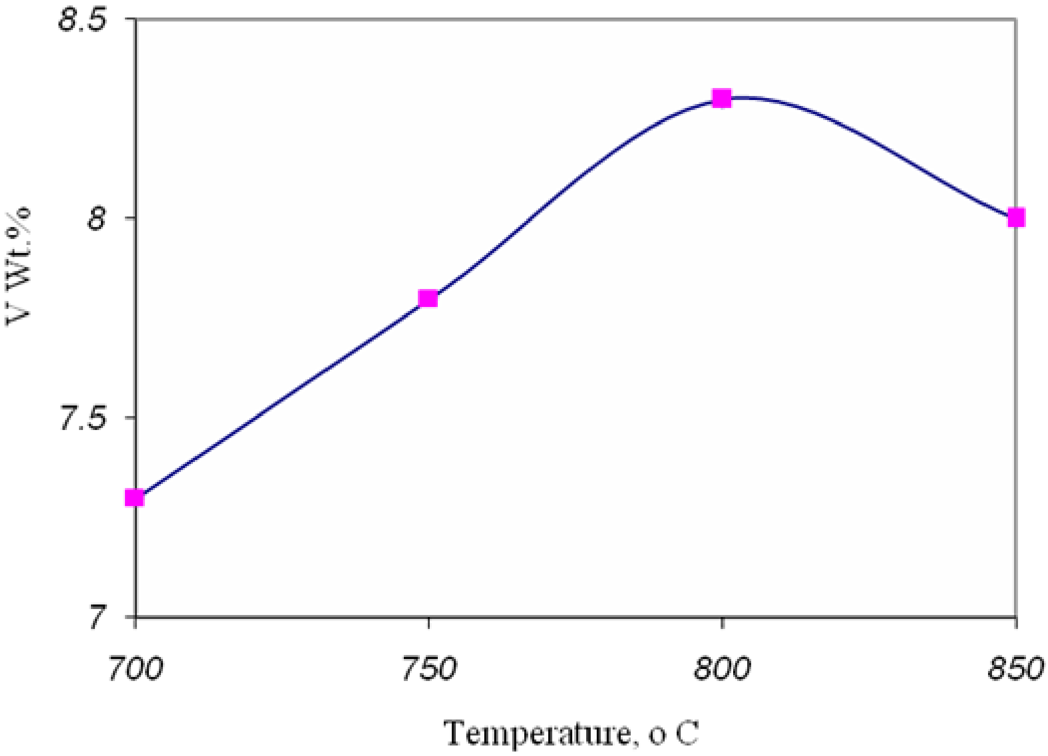

Figure 2. Effect of temperature on the recovery of vanadium in the produced composite.

Effect of Temperature

The effect of bath temperature on the recovery of vanadium in the produced Al-Al_3V composite was investigated in the range from 700 to 850 ^oC as show in Figure (2). From this Figure, it can be noticed that there is a linear increase of the vanadium contents in the produced composite in the range from 700 to 800 ^oC. This is due to the increasing fluidability of the bath which made a chance for insitue formation of Al_3V. But when the bath temperature increased to more than 800 ^oC, the vanadium contents in the produced composite are decreased due to the loss of some vanadium by oxidation.

Heat: Figure 3 illustrates the DTA analysis for mixture of powdered V_2O_5 and Powdered Al, , from this figure it can be noticed that the exothermic peak appears at about 600 ^oC. This means that the reaction 1 started exothermically at about 600 ^oC. The mechanism of formation of Al_3V intermetallic compound insitue within the molten Aluminium is according to the following reactions:

$3V_2O_5$(Powder) + 10 Al (Powder) $\rightarrow$ 6V (active) +$5Al_2O_3$ + Heat (1)

V (active) +3 Al (molten) $\rightarrow$ Al_3V(in situe in Aluminium matrix) (2)

The reaction 1 is exthothermic reaction leading to rise in the bath temperture from 850 ^oC to about 1000 ^oC. But reaction 2 forms Al_3V insitu the molten Aluminium, the increase in bath temperater more than 1000 ^oC makes some loss of vanadium in the produced composite owing to oxidation affinity.

The effect of Al /V_2O_5 weight ratio (R)

The effect of Al /V_2O_5 weight ratio (R) was studied from 0 to 0.75, as shown in Figure 4. The recovery of vanadium in the produced Al-Al_3V composite increased linearly with increasing R in the range of 0 to 0.5. This is due to the increase in vanadium oxide quantity that was added in the reaction bath. But, a little slight increase in vanadium recovery in the produced Al-Al_3V composite is due to increasing R in the range of 0.5 to 0.75. According reaction 1, the equivalent ratio of Al /V_2O_5 weight ratio (R) is closed to 0.5. So, at R = 0.75, the amount of powdered Al is more than the amount of V_2O_5, so the all V_2O_5 is reduced to active vanadium according to reaction 1 and the reminder powdered Al helps to form Al_3V intermetallic compound insitue formation within the molten Aluminium, lead to increase the efficiency of vanadium recovery.

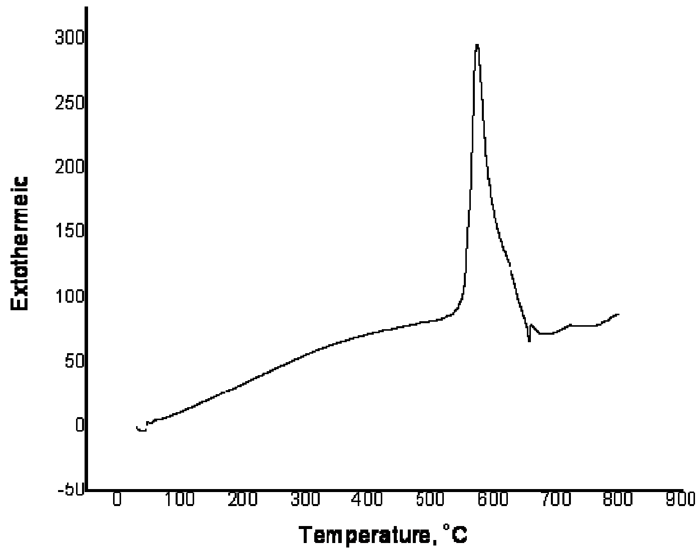

Figure 3. show the DTA analysis for V_2O_5 and Al (Powder).

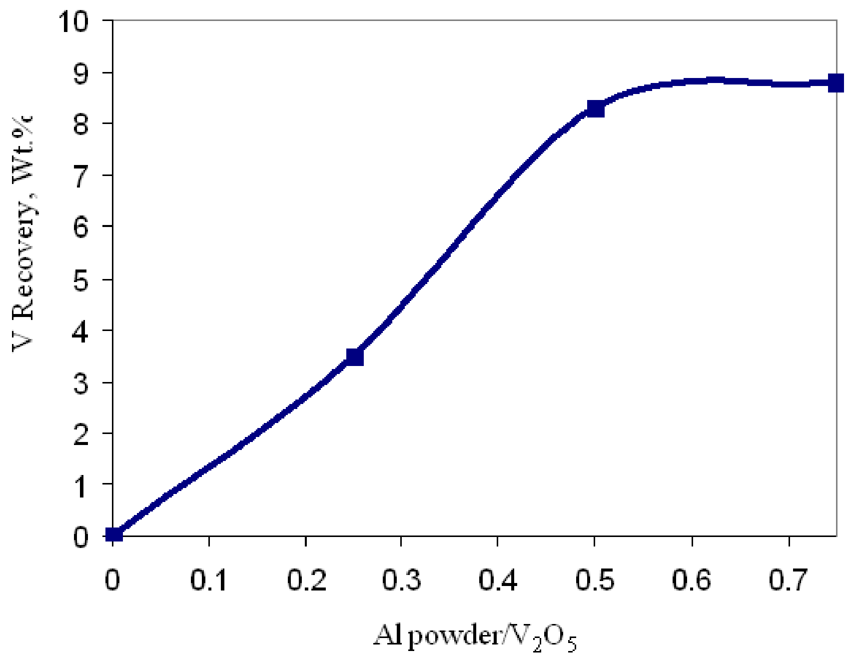

Figure 4. Effect of Al powder/V_2O_5 on the recovery of vanadium in the produced composite

Characterization of the Al_3V intermetallic compound in produced composite

The X-ray diffraction analysis for the produced Al-Al_3V composite containing 8.3%V was carried out from 20 to 70 degree. Figure 5 shows the XRD pattern, the phase identifications for this pattern indicated that only two phases appeared; pure Al and Al_3V. According to the Al-V phase equilibrium diagram Figure 6, the compounds $Al_{21}V_2$, $Al_{45}V_7$ and $Al_{23}V_7$ are metastable over temperature 736°C (1009K). But the compound Al_3V is a stable compound until 1420 °C(1693K) as shown in Figure 6, also from this Figure it can be noticed that the composition of Al_3V contains about 39 Wt.% V (25% at%) and 61 Wt.% Al (75% at%) (Murray, 1989; Kostov et al., 2006).

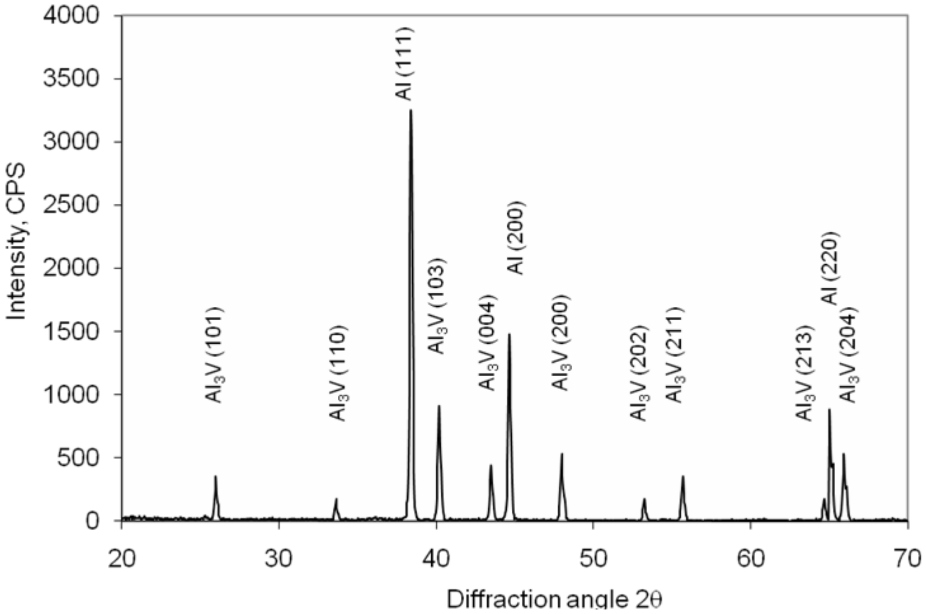

Figure 5. X-ray diffraction pattern for the produced composite containing 8.3 Wt.% V

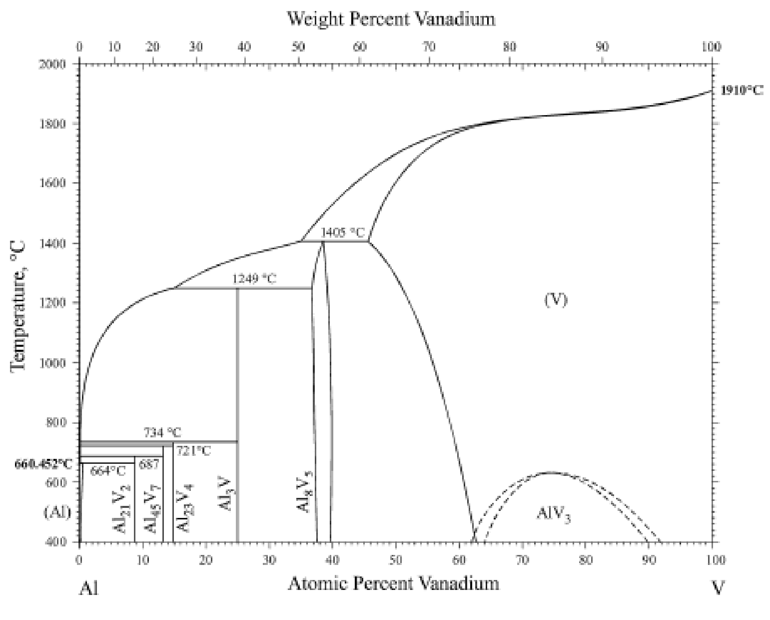

Figure 6. Phase diagram of Al-V system (J. L. Murray, (1989); A. Kostovet al, 2006).

From X-ray diffraction data obtained using PDF2, programs, the crystal structure of Al_3V is body-centered tetragonal tI8 space group is (I4/mmm) lattice parameters are: a, b is 3.78 Angstroms, and c is 8.322 Angstroms, and the density is 3.65.

Figure.7 shows SEM micrographs of the prepared composite containing 8.3%V by. From this figure, it can be noticed that a homogenious distribution from (light gray phase) nodule like dispersed with in dark gray matrix. The point analysis of the light gray phase at point 1 using energy disparsive spectrometer (EDS) as shown Figure 8 is 39.76 Wt% vanadium and 60.24 aluminium. From Al-V phase equlibrium diagram, this analysis is much closer with the composition of the verticle line which represents Al_3V intermetallic compound. This result is confirmed with the result of XRD analysis. While the

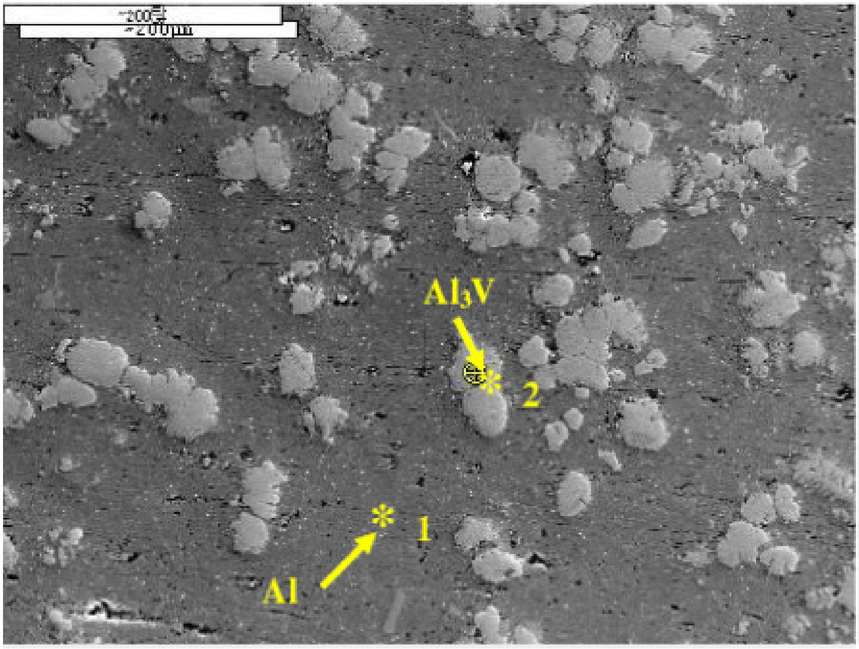

Figure 7. SEM micrograph for the produced Al_3V indicate two phases Al matrix at point 2 and Al_3V at point 1

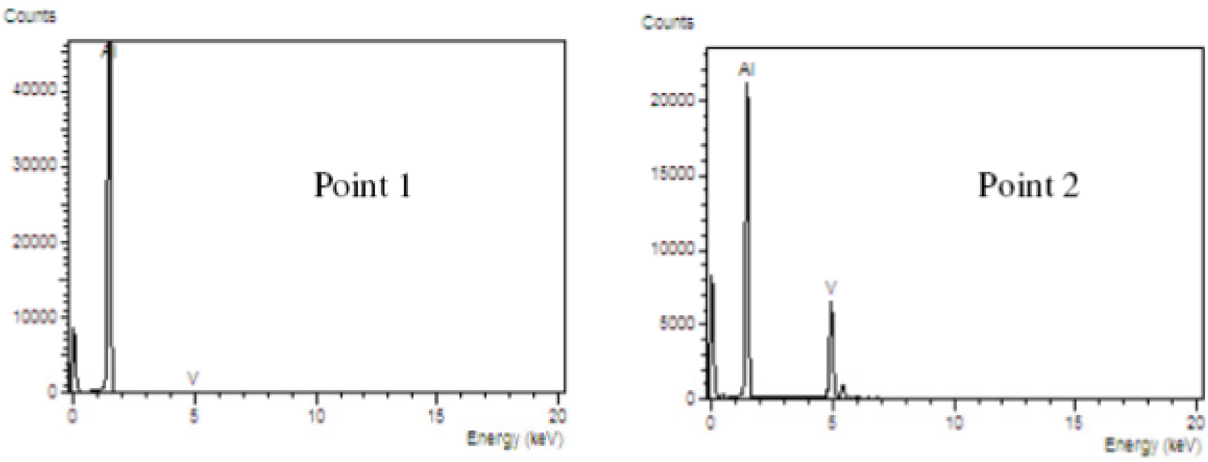

Figure 8. EDS analysis for the produced Al_3V at point 1, 2 indicated above in Figure. 6

point analysis of the dark gray phase at point 2 using EDS Figure 8 is 0.3 Wt% vanadium and 99.7 aluminium. This means that the light gray phase is Al_3V and the dark gray phase is aluminium matrix.

Figure 9 shows the backscattered electron SEM micrographs for the prepared Al-Al_3V composite. From this Figure it can be noticed that a homogeniously distributed Al_3V compound (white phase) appears like cotton within the aluminium matrix (black). This is confirmed with the mentioed above in bath XRD, and SEM. The distribution of Al_3V within Al matrix may reinforce the matrix and imrove the mechanical properties.

Figure 10 indicates the X-ray mapping images and the distribution of Al, and V in the Al-Al_3V composite prepared at 800 ^{o}C and R is 0.5. From this figure it can be seen that SEM photograph contaning Al_3V within the aluminium matrix as mentioned in Figure 10 (a). but the bottom micrograph Figure 10 (b) indicates only Al particles or atom (gray coulor) and the place of vanadium

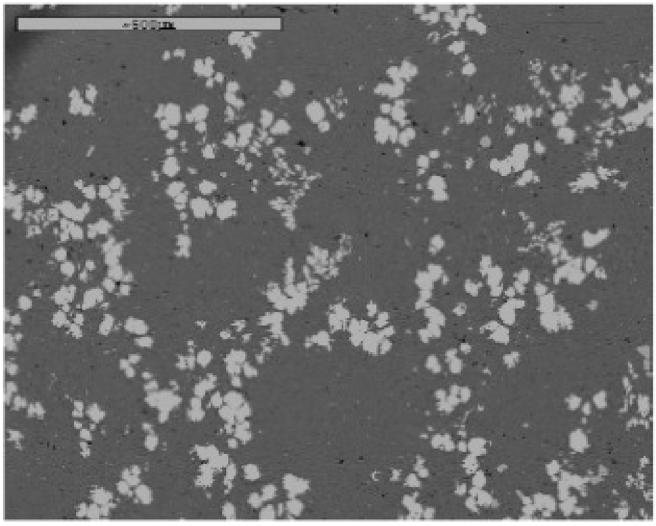

Figure 9. Backscattered electron SEM micrographs of the prepared Al_3V

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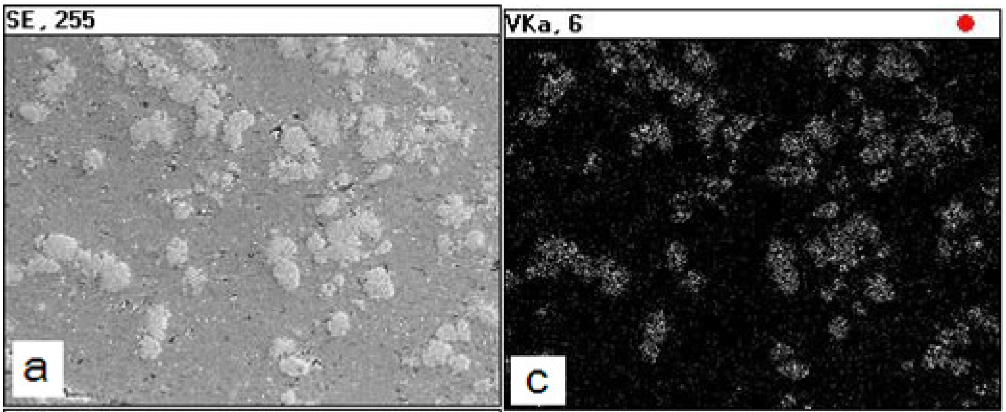

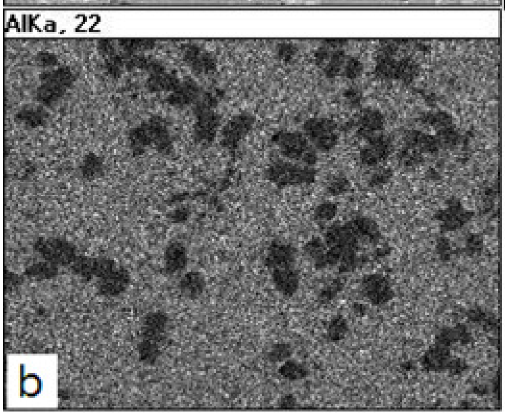

Element	Wight%	Atomic %
Al	91.69	95.42
V	8.31	4.58

d

Figure 10. X-ray mapping images indicates the distribution of Al, and V in the prepared Al_3V 100 µm

particls (black) like shadow. The right micrograph Figure 10 (c) indicates only vanadium particles (white coulor) and the place of aluminium particles black color. It can be conclouded that the white gray nodules is a mixture of

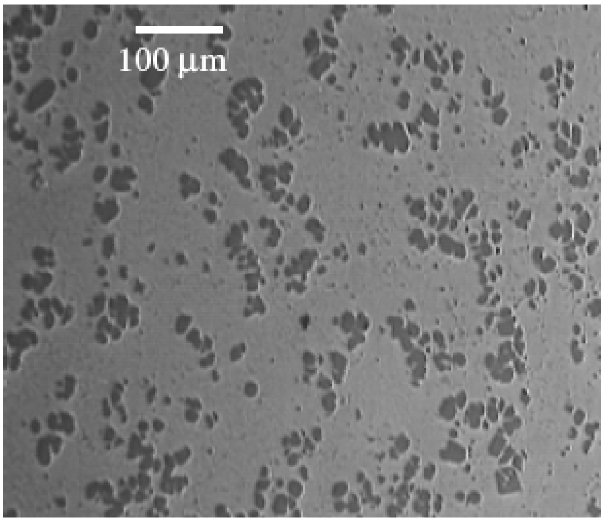

Figure 11. Light microscopy image shows the distribution of Al, and V in the prepared Al_3V

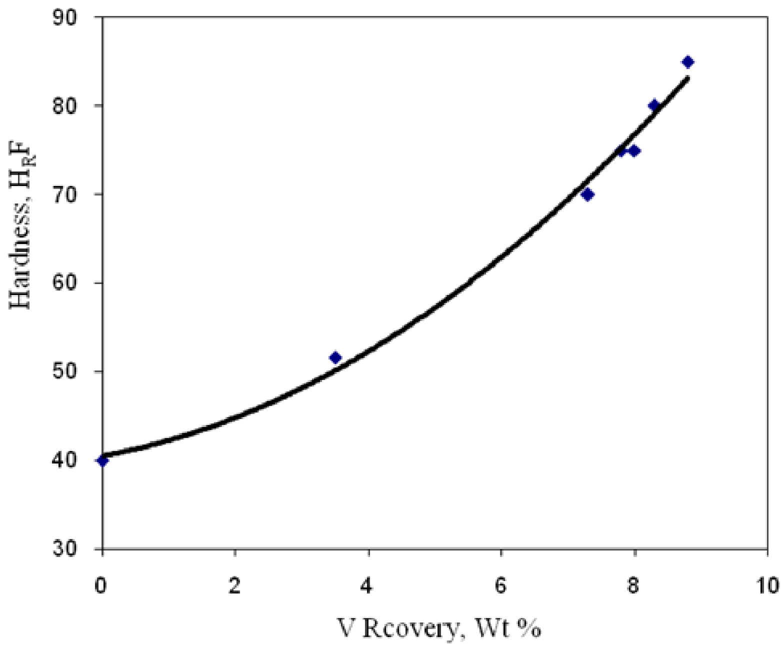

Figure 12. Effect of V recovery on the Hardness H_RF of the prepared composite reinforced by Al_3V intermetallic

alminium particles and vanadium particles form Al_3V and the dark gray is aluminium matrix, this is confirmed with the results mentioned before. The all matrix analysis using EDX proved that the analysis of aluminium is 91.69 wt%Al and the vanadium is 8.31 Wt% V as shown in Figure 10 (d).

Also, Figure 11 shows light microscopy image indiates the distribution of Al, and V in the prepared Al_3V. the same results are obtained from this figure, the nodules Al_3V (black) disparsied in aluminium matrix dark gray and this is more evedance to indicate that the phase appear in the aluminium matrix is absolutly Al_3V inermatallic compound and may reinforce the aluminium matrix.

Figure 12 shows the relation between hardness (H_RF) of the prepared composites reinforced by Al_3V intermetallic and vanadium recovery. The hardness is significantly increased as vanadium recovery increased from 0 to 8.8 V%. This increasing may be because many Al_3V intermetallic compound was formed. which is the main reinforcements in the Al- Al_3V composites. The releation between hardness (H_RF) of the prepared composites and vanadium recovery can be obtained from the following equation:

$$H_RF = 0.39\ V^2 + 1.37\ V + 40.47 \qquad (3)$$

From the previous results, it can be said that the new technique for preparing Al-Al_3V composites was successful. The presence of the Al_3V intermetallic compound in the aluminium matrix enhanced and improved the mechanical properties of the prepared composite.

Conclusions

In this work, a developed method for preparation of Al-V composite from reduction of V_2O_5 by powdered aluminium within a bath of molten aluminium has been proposed. The main results were concluded as follows:

1. The mechanism of the Al_3V intermetallic compound insitue formation within the molten Aluminium is that , firstly the aluminium powder reduced the V_2O_5 to librate vanadium ions, the later reacted with molten aluminium to form Al_3V intermetallic compound insitue.
2. The recovery of vanadium in the prepared composite is cosed to 8%V and The maximium efficiency of the V recovery up to 80% has been achieved.
3. According to DTA analysis, the reduction reaction is an exothermic reaction at temperature more than 600°C and the uncontrolled bath temperture reached up to 1000°C.
4. From XRD, two phases only appeared; pure Al and Al_3V. Also, according to the Al-V phase equilibrium diagram, the compounds $Al_{21}V_2$, $Al_{45}V_7$ and $Al_{23}V_7$ are metastable over temperature 736°C (1009K). But the compound Al_3V is a stable compound until 1420 °C(1693K, also it can be noticed that the composition of Al_3V contains about 39 Wt.% V (25% at%) and 61 Wt.% Al (75% at%).
5. The SEM indicated that a homogenious distribution from (light gray phase) nodule like dispersed within dark gray matrix. The point analysis of the nodules using energy (EDS is 39.76 Wt% vanadium and 60.24 aluminium. This analysis is much closer with the composition Al_3V intermetallic compound. This means that the light gray phase is Al_3V and the dark gray phase is aluminium matrix.
6. Although the Al_3V intermetallic compound is an unknown compound in open literature, , it has proved certainly by introducing many evidence using XRD, SEM, EDS, DTA and X ray mapping that the formed compound is Al_3V intermetallic compound.
7. The presence of this compound in the aluminium matrix enhanced and improved the hardness of the prepared Al-Al_3V composite.

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