PUBLICATION PAPER

THE EFFECT OF ALLOY COMPOSITIONS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF CERAMIC MATRIX COMPOSITE FORMED BY COMBUSTION SYNTHESIS

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Arranged by:
YUDHA RAHMAN AWALLU
D200102004

MECHANICAL ENGINEERING DEPT. INTERNATIONAL PROGRAM IN AUTOMOTIVE/MOTORCYCLE ENGINEERING
UNIVERSITY MUHAMMADIYAH OF SURAKARTA
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Written by:

Name : **Yudha Rahman Awallu**

NIM : **D200 102 004**

Has approved and legalized on:

Day : Saturday

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Approved to be examined by Supervisors Team:

Supervisor II	Admitted by: Secretary of International Program,
(Wijianto, ST. M. Eng. Sc.)	(Wijianto, ST. M. Eng. Sc.)

THE EFFECT OF ALLOY COMPOSITIONS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF CERAMIC MATRIX COMPOSITE FORMED BY COMBUSTION SYNTHESIS

Yudha Rahman Awallu

Automotive/Motorcycle Engineering Department

Muhammadiyah University of Surakarta

Jl. Ahmad Yani Tromol Pos 1 Pabelan Kartasura 57102

Email: yudha.rahman16.yr@gmail.com

ABSTRACT

In this research the purpose its to showing the effect of alloy compositions. This research was done by using powder form materials such as C, Al, and TiO_2. By used formula $3TiO_2$ + (4+X) Al + 3C → 3TiC + $2Al_2O_3$ + (X) Al, the powders reacted also with excess Al to see the different of effect that was done by combustion synthesis.

The results from the research is to showing the efect of alloy by excess Al in the reactant composition, that excess Al from 20%, 40%, 60%, 80%, and 100%. The synthesis process will be done by using Arc Flame and argon, argon to minimize the oxidation from oxygen. The product from the research is Al_2O_3 aliminium alloys are alloys in which aluminium (Al) is the predominant metal. The typical alloying elements are copper, magnesium, silicon, tin, and zinc. About 85% of aluminium is used for wrought products, for example rolled plate, foils, and extrusion. To know what the effect from the excess of Al to the reactant, in this research the test will be done by using XRD test, SEM, and microhardness. XRD test used to know the phase of the product, in the other way SEM test used to show the composition of the product after burning process, and the last one microhardness test it's done to know the physical properties.

Keywords: Combustion synthesis, Aluminium alloys, XRD, SEM, Microhardness.

Background

Ceramics have attracted wide interest as advance materials for high temperature applications due to their advantages such as high modulus, high hardness, high melting temperature, and high corrosion resistance. Alumina ceramic systems such as $TiC–Al_2O_3$ have also been widely found in high temperature applications since they have high hardness and low density. It was also reported that the binary ceramic composites of $TiC–Al_2O_3$ could improve the fracture toughness of the individual ceramic materials, either TiC or Al_2O_3.

The combustion synthesis (CS) process offers significant advantages to fabricate ceramic materials since it produces high exothermic reaction and a short processing time.

Problem Statement

The problem how is to produce an aluminium alloy material using combustion synthesis proces and understand the effect of different reactant compositions on the product.

Objective of Study

The objectives of this final project research are as follows:

1. To produce an aluminium alloy material by combustion synthesis process.
2. To observe the composition of synthesized product.
3. To evaluate the mechanical properties of the synthesized product.

Literature Review

Aliminium alloys are alloys in which aluminium (Al) is the predominant metal. The typical alloying elements are copper, magnesium, silicon, tin, and zinc. About 85% of aluminium is used for wrought products, for example rolled plate, foils, and extrusion.

Combustion Synthesis

The Science of Nanomaterials is proving to be one of the most attractive and promising fields for technological development in this century. One of those process is synthesis material with chemical reaction between 2 or more material become one structure of material, by ceramic method is controlled by the diffusion of atoms and ionic species through reactants and products and thus requires repeated grinding, pelletizing, and calcinations of reactants at high temperatures.

Ignition Techniques

a. Microwave

The application of microwave energy to the processing of various materials such as ceramics, metals and composites offers several advantages over conventional heating methods.

Microwave heating is fundamentally different from the conventional one in which thermal energy is delivered to the surface of the material by radiant and/or convection heating that is transferred to the bulk of the material via conduction see figure 1.

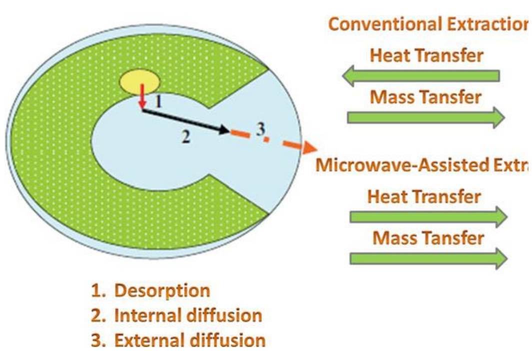

Figure 1. Basic heat and mass transfer mechanisms in microwave and conventional extraction

b. Electric Current Activated/assisted Sintering (ECAS)

The ECAS process it simultaneously applies an electric current along with a mechanical pressure in order to consolidate powders or synthesize and simultaneously densify specific products with desired configuration and density. The applied electric current and mechanical load may be constant throughout the sintering cycle or may vary during the selected densification stages see figure 2

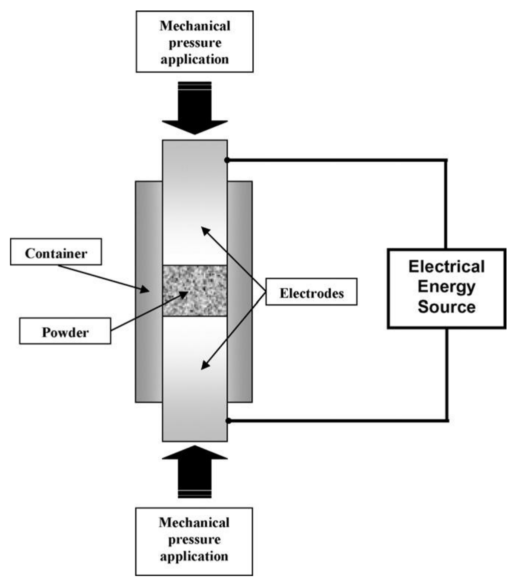

Figure 2. Schematic representation of the ECAS

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Research Methodology

Experimental investigations to produce aluminium alloy using the combustion synthesis of the powder mixture are carried out using the following procedures:

a. Preparation of starting material

In this work, powder was used as the reactant based on the consideration that powders offer a possibility to produce complex geometries of product and provides “near net shape” forming which can eliminate machining operations. The powders used in this work are aluminium (Al), titanium oxide (TiO_2), and carbon black (C).

b. Sample composition

The reactant powders used for the combustion synthesis are weighed according to the mass fraction of each element as described in the chemical reaction in Eq. 1.

$$3TiO_2 + (4+X)\ Al + 3C \rightarrow 3TiC + 2Al_2O_3 + (X)\ Al \quad (1)$$

c. Measuring weight of materials

Measuring each material it so important to achieve good mixing, each material was in powder form this was to make easy contact ignition process.

d. Powder mixing

The main intention of powder mixing is to achieve a mixture which is as homogenous as possible since the mixture homogeneity significantly affects the microstructure and properties of the product. The mixing of reactants is carried out by crushing approximately 10 grams in a ceramic mortar for about 20 minutes.

Figure 3. Ceramic mortar

Then we take 5 samples of the powder mixture, each is 1gr and add Aluminum to each sample (20%, 40%, 60%, 80%, 100%) respectively of the main sample percentage.

e. Powder compaction

The compaction of the mixture is performed by applying pressure to produce pellets with a certain density and thickness. In this work, the compaction was carried out using a cylindrical die and punch of steel. The inner diameter of the die is 16.0 mm. Before the compaction process, the entire wall surfaces of the punch and die are cleaned and must be free from dust and any grease in order to reduce friction during the compaction. When the mixture has been filled in the dies, the compaction is done using a torque key.

f. Reaction chamber

The combustion synthesis is carried out in a sealed reaction chamber in order to control the atmosphere during the process. Generally, combustion synthesis can be conducted in a vacuum, argon, or nitrogen inert gas atmosphere to minimize the oxidation.

g. Ignition techniques

The main goal of selecting the ignition method in the present work is to obtain fast heating of the sample to initiate the ignition of combustion synthesis.

In this process the pellet should be burn by Arc flame with argon to protect the surrounding air from oxidation.

Figure 4. Arc flame equipment

Microstructure

The results on this research is to create material Al_2O_3 by combustion synthesis where, is done by several test. This path to show that the research is complete, the test materials are XRD, SEM, and micro hardness.

a. X-ray diffraction (XRD)

X-ray Diffraction is a tool to identifying the atomic in material of a crystal, this crystalline atoms cause a beam of incident X-ray to diffract into many specific direction. By measuring the angles and intensities of these diffracted beam, a crystallographer can produce a three dimensional picture of the density of electrons with the crystal. Form this electron density, its mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder and various other information.

b. Scanning Electron Microscopy (SEM)

The scanning electron microscope is one of the most way tools that available for examination and analysis of the microstructure morphology and chemical composition characterizations. SEM is used for observation of specimen surfaces, when the specimen is irradiated with a fine electron beam, secondary electrons are emitted from the specimen surface see figure 5.

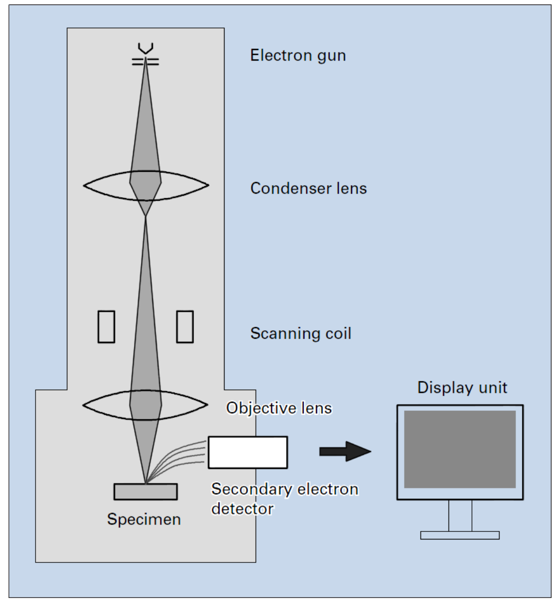

Figure 5. SEM basic construction

c. Microhardness

In metal material crystallization is a small specimen that sticks together and become one unit that we know as material. A material produced by specific reaction where, here because combustion synthesis, the defect from the product could be happen by any errors in the process. By micro hardness test the properties of the material can be finding especially in physical properties.

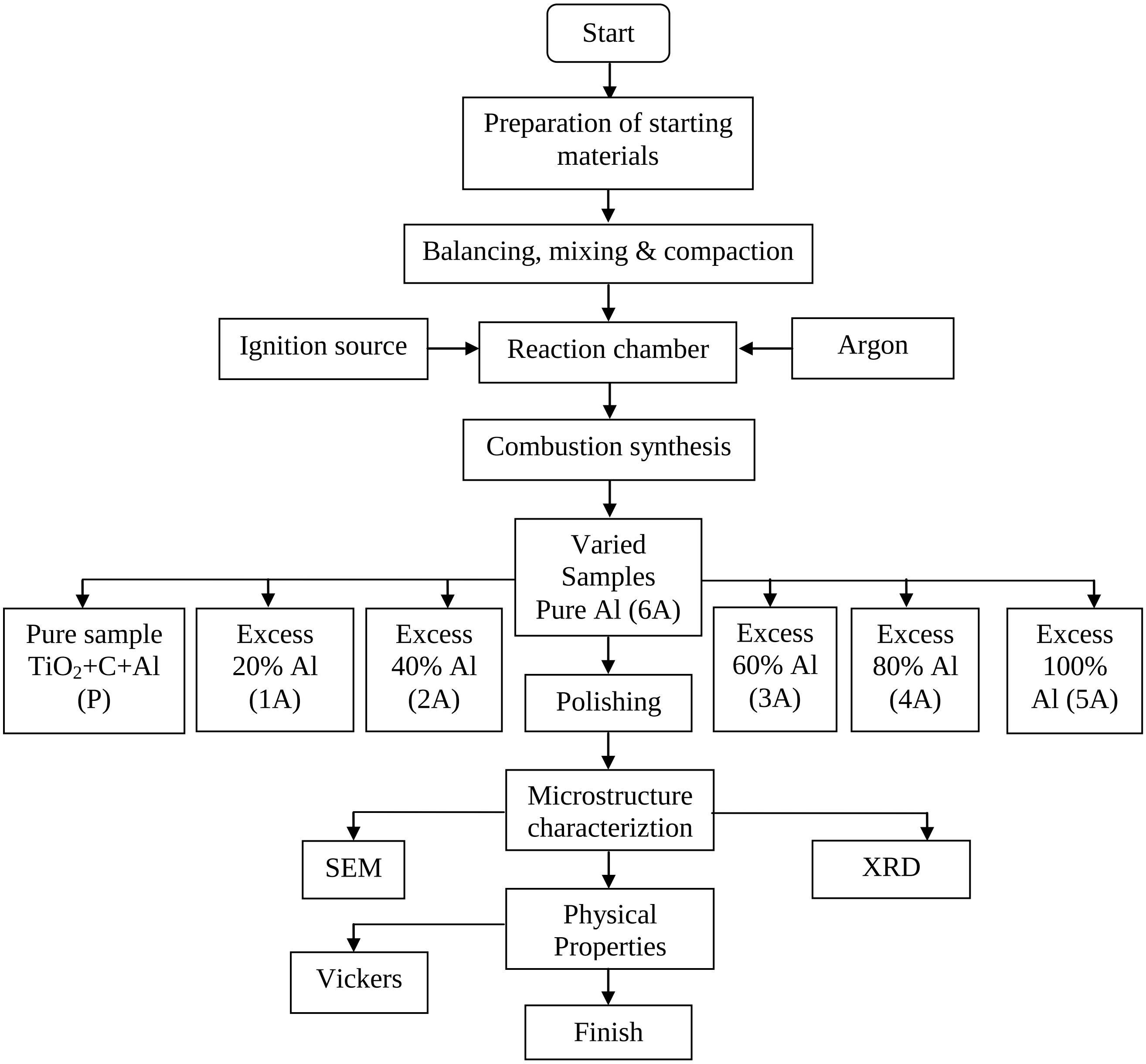

Figure 6. Research flow chart

Results and Discussion

a. Calculation of the reactant mass

Measuring the mass of each material is important to achieve good composition of the reactant. Each material used in the present work was in powder form.

This was to make easy contact between the particles for the purpose of ignition process. Measuring the weight of each element was carried out by the calculations below:

$3TiO_2 + (4+X)\ Al + 3C \rightarrow 3TiC + 2Al_2O_3 + (X)\ Al$

Mass of TiO_2 = 3 (47.90 + (2x 15.99)) = 239.694 gram

Mass of Al = 4(26.9815) = 107.926 gram

Mass of C = 3 (12.011) = 36.033 gram

Theoritical Total Mass = 383.653

Practical Total Mass is 1 gram, so:

Mass Of TiO_2 = $\frac{239.695}{383.653}$ X 1 = 0.625 gram

Mass of Al = $\frac{107.926}{383.653}$ X 1 = 0.281 gram

Mass of C = $\frac{36.033}{383.653}$ X 1 = 0.094 gram

Then the table 1 shows the variations of amount excess Al.

Table 1. Amount of excess Al

Samples	**Excess Al**	**Amount of Excess Al (gram)**	**Description**
1A	0.2 X 1 gr	0.2	-
2A	0.4 X 1 gr	0.4	-
3A	0.6 X 1 gr	0.6	-
4A	0.8 X 1 gr	0.8	-
5A	1 X 1 gr	1	-
6A	-	-	Pure Al
P	-	-	Standard composition (TiO_2+C+Al)

b. Combustion synthesis process

In this step, the specimen will be burning by arc flame with argon. The argon gas was used to avoid the combustion process from the oxidation. As we know that there were 5 specimens in which each specimen has different mass of excess Al. The mass of Al excess was in the range of 20% to 100%.

Figure 7 shows the synthesized product of sample 1A.

Figure 7. Synthesized product of sample 1A

Microstructure characterization

a. Phase identification of product using XRD

By this test the crystal of the material can be known by diffraction of the beam. The material that uses in this test should be in the crystal structure because if the material doesn't have the crystal it can't work. So the ignition process should be done well, if not the XRD spectra of the material will not appear. By using the XRD machine we know the XRD spectra. In this test we have 3 samples where XRD spectra of the sample of 1A, 2A, and the pure sample are given in figure 8 to 10.

Figure 8 above shows the signal of crystal from the pure sample (P). As we can see varieties of Theta there are 4 signals that showing Al_2O_3. The highest signal is with number of counts 6 with theta 37.60, and then the second is with number of counts 3 at theta 42.82, the last one theta 46.3 and 67.3 with number of counts 1.

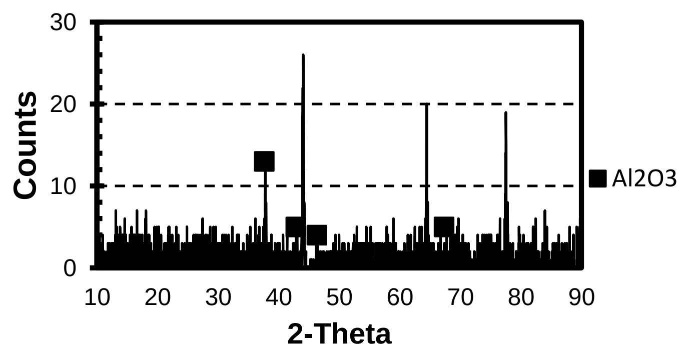

Figure 8. XRD phase sample P ($3TiO_2/4Al/3C$)

Figure 9 shows the results of sample 1A which composed of $3TiO_2/4Al/3C$ with 20% of Al excess. It can be seen in the graph that it shows the XRD spectra from the material. The graphic line tells us that the composition of the compound Al_2O_3 are at theta 15.42 with counts 6, at theta 33.28 with counts 2 and at theta 35.74 with counts 8. Because in this sample was excess al 20% so there are some Al showing in this XRD spectra there are in theta 38.48 with counts 4, theta 44.74 with counts 4, theta 65.14 with counts 2, and the last one with theta 78.22 and 82.44 with counts number 1 and 2.

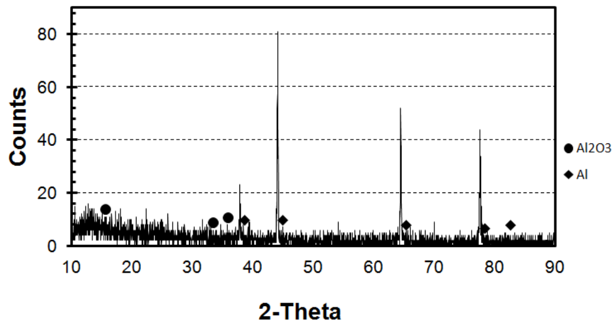

Figure 9. XRD phase sample 1A

Figure 10 shows the XRD data that was taken from sample 2A. The results give data that the ignition process was success, because the XRD machine can read the crystal from the sample, the composition of the compound Al_2O_3 on theta 15.44 with counts 5, theta 19.46 with counts 6, theta 32.42 with counts 4, and the last in at theta 36.96 with counts 2. This sample was excess al 40% so there are some Al showing in this XRD spectra there are in theta 38.48 with counts 2, theta 44.74 with counts 2, next at theta 65.14 with counts 3, the last at theta 78.2 and 82.44 with counts 4 and 2.

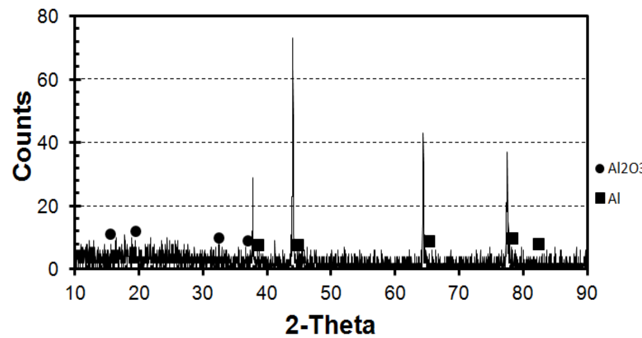

Figure 10. XRD phase sample 2A

b. Microstructure (SEM)

From table 2 we can see the composition of pure sample after burning process. The compound of Al_2O_3 was showing and processing so well with amount 31.31 %, and there is a new compound the reacted into the specimen this could be happen in the ignition process the compounds is CaO with amount 0.41% this compound is doesn't take much.

Table 2. Composition of sample pure after burning

Compound	Mass (%)
C	15.86
Al_2O_3	31.31
CaO	0.41
TiO_2	52.42
Total	**100**

The table 3 shows the composition of sample 1A after burning process, by excess of Al 20% or 0.2 gram. The mass of Al_2O_3 is 52.8 % from the total weight. It showing the reaction was success. But as we can see TiO_2 doesn't reaction so well, see (table 3.1) as we can see the mass TiO_2 was 62.47% before the burning process and then after the process is 30.47% it showing half of the material doesn't reaction. Also there is additional element Nitrogen (N) it might be from the ignition process where, the ignition process has done by using steel plate to flow the electric charge.

Table 3. Composition of sample 1A after burning

Compound	Mass (%)
C	12.50
N	4.23
Al_2O_3	52.8
TiO_2	30.47
Total	**100**

Table 4 shows the composition of sample 2A after burning process, by excess of aluminum 40% or 0.4 gram, amount of Al_2O_3 was increasing where, it reach number 83.85% of mass. As we can see it is higher than sample 1A with mass just 52.8%. Almost all reaction is dominated by Al_2O_3.

Table 4. Composition sample 2A after burning

Compound	Mass %
C	7.04
N	8.25
Al_2O_3	83.85
CaO	0.34
TiO_2	0.52
Total	**100**

c. Microhardness

From the table 5 as we can see the results of each point was different, those variation happen because the position of the force didn't in the same place, this way to make the results of the test more precise. By taking the average from the 3 test we can assume the truth result.

From the table 5 we can see that sample 1A is the highest result with number 1241.867 HV where, the specimen with excess Al 20% shows that is greater of hardness by excess 20% Al. But the sample 6A is the lowest with 33.27 HV this sample is pure Al this make assume that Al_2O_3 is better than pure Al.

Table 5. Hardness test results

Specimen	Applied point (HV)			Average (HV)
	Point 1	Point 2	Point 3	
1A	1602	1872	251.6	1241.86
2A	194.6	96.8	102.2	131.2
3A	224.6	22.3	132.33	126.33
4A	98.2	20.5	188.6	102.43
5A	74.9	196.4	71.7	114.33
6A	38.5	24.4	36.9	33.27

The figure 11 shows the result of hardness test, as we can see and discuss before specimen 1A was the greater physical properties with number 1241.867 HV, and specimen 6A is the lowest result with 33.27 HV. Look to the graph the best excess of Al to the specimen is in sample 1A by excess 20%, if we put more the Al then the hardness of the material will be decrease as the graph showed.

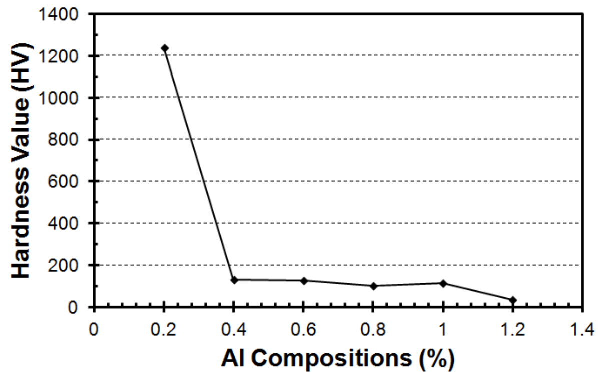

Figure 11. Graph of microhardness test result

Conclusion and Suggestion

According to the research and based on the experiment, we may conclude the following:

1. The experiment succeeded by producing Al_2O_3 and this element appeared as the prediction. However, TiC cannot be detected.
2. By increasing the excess of Al to the sample produces more Al_2O_3.
3. The physical properties are greater when excess Al 20% than pure Al, but by adding more Al to the reaction will decrease the hardness of material.

In this experiment the researcher has done the research, and there are some things that should be understood in the process of the research, there are some suggestions to be done in the future to make the research much better which are:

1. In the ignition process or burning process, a combustion chamber is to be used to protect the ignition process from oxidation and other surrounding elements.
2. Always use gloves and a mask in every process to protect from dangerous affects.
3. In the mixing process the materials should be mixed well, then predict how long is the time for the best mixing process
4. Clean all the tools before and after using such as, glass stirrer, ceramic mortar, dies, and beaker glass.
5. Try the combustion synthesis with electric charge or microwave to get the best results.

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