CHAPTER 1
INTRODUCTION

1.1 Background

The keris is synonymous with the Malay culture and way of life. The double-edged dagger is unique because it is only found within the Malay Archipelago. Almost all cultures around the world have swords. What makes the keris one of a kind are the details found at the base of the blade; details that exist in no other swords. At its best, the keris represents the highest level of Malay creativity. A long time ago, it is used to complete the Malay attire. Walking around without the keris for a Malay men back then was like to walk around naked. In modern Malaysia, it now has become a royal sovereign symbol and prized works of art to be collected. The keris as a work of art, portrays the highest quality of workmanship.

Keris is a special weapon as it was made from Damascus steel. Damascus steel is a hot-forged steel that been used in sword making from about 1100 to 1700 AD. Damascus steel was of its legendary sharpness and strength, and were apocryphally claimed to be able to cut through lesser quality steel and even rock. The technique used to create original Damascus steel is now a matter of historical conjecture. When forming a batch of steel, impurities are added to control the properties of the resulting alloy. In general, one could produce an alloy that was hard yet brittle at one extreme by adding up to 2% carbon, or a higher level of toughness yet ductile and malleable at the other, with about 0.5% carbon. The problem for a sword smith is to create steel which strikes the perfect balance between hard and tough - hard, so as to hold an edge once sharpened, but tough so it would not shatter when hitting other metal in combat.

In nowadays application, Damascus steel had been used for high quality castings for the most challenging wear-resistant applications. It has been used widely in mining and railroad, steel mills, seamless tube and pipe and also oil and gas industry. This project will help to reveal the truth back on the history of the keris blade by studying the

microstructure of the keris blade itself. Besides that, this project also is to find out the mechanical properties of the keris blade that origin from Peninsular Malaysia.

In order to study on the microstructure on the keris blade, Nondestructive tests (NDT) will be used throughout this project. NDT plays a crucial role in ensuring cost effective operation, safety and reliability of plant, with resultant benefit to the community. NDT is used in a wide range of industrial areas and is used at almost any stage in the production or life cycle of many components. For implementation of NDT it is important to describe what shall be found and what to reject.

As we know, keris blade had been used as a defend and attack weapon in the past. So, the main characteristics of a good defend and attack weapon is it must be high in strength and also hard enough to be able to stab at the opponent. It is important for us to look and study into keris mechanical properties to reveal the truth on this weapon mechanical properties such as its hardness.

1.2 Problem Statement

1.2.1 Keris had been used for a long time ago but until now there is no proper study had been establish yet on the microstructure of the keris blade that origin from Peninsular Malaysia.

1.2.2 Furthermore, there is also no proper study had been done on the mechanical properties of the keris blade.

1.2.3 Keris making process had been a secret to the public and few know on the science and engineering in making the keris.

1.3 Objective

1.3.1 To study on the microstructure on traditional keris blade origin from Peninsular Malaysia

1.3.2 To study on the mechanical properties of the keris blade origin from Peninsular Malaysia

1.4 Scope of Study

1.4.1 To run various Non-Destructive Test such as Optical Microscopy and X-ray Diffraction (XRD) on two different keris blade which is from Kuala Kangsar and Perlis

1.4.2 The scope of the Non-Destructive Test is to determine the microstructure properties of the welded Damascus steel use at keris blade

1.4.3 To run a microhardness test on the keris blade which will be used to find out the mechanical properties (hardness) of the keris blade

CHAPTER 2

LITERATURE REVIEW

2.1 Microstructure

Many times, the physical properties and, in particular, the mechanical behaviors of a material depend on the microstructure. Microstructure is subject to direct microscopic observation, using optical or electron microscope. In metal alloys, microstructure is characterized by the number of phases present, their proportions, and the manner in which they are distributed or arranged. The microstructure of an alloy depends on such variable as the alloying elements present, their concentration and the heat treatment of the alloy (i.e., the temperature, the heating time at temperature and the rate of cooling to room temperature)

After appropriate polishing and etching, the different phases maybe distinguished by their appearance. For example, for a two-phase alloy, one phase may appear light and the other phase dark. When only a single phase or solid solution is present, the texture will be uniform, except for grain boundaries that maybe revealed.

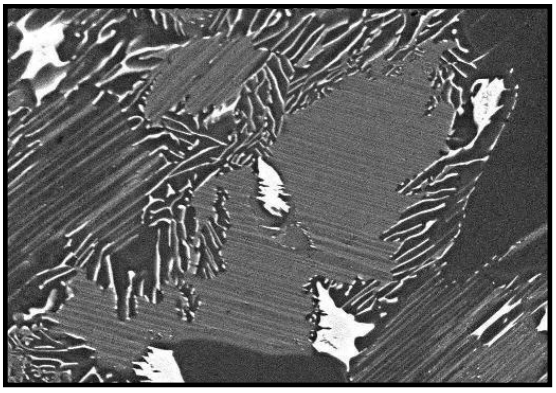


Figure 1: Example of microstructure of a TiAl alloy aged at 800 ^{o}c [1]

2.2 Microscopic Examination

On occasion it is necessary or desirable to examine the structural elements and defects that influence the properties of materials. Some structural elements are of macroscopic dimensions; that is, they are large enough to be observed with the unaided eye. For example, the shape and average size or diameter of the grains for a polycrystalline specimen is important structural characteristics. Macroscopic grains are often evident on aluminum streetlight posts and also on highway guardrails. Relatively large grains having different textures are clearly visible on the surface of the sectioned lead ingot. However, in most materials the constituent grains are of microscopic dimensions, having diameter that may be on the order of microns, and their details must be investigated using some type of microscope. Grain size and shape are only two features of what is termed the microstructure.

Optical, electron and scanning probe microscopes are commonly used in microscopy. These instruments aid in investigations of the microstructural features of all material types. Some of these techniques employ photographic equipment on conjunction with the microscope; the photograph on which the image is recorded is called photomicrograph. In addition, many microstructural images are computer generated and/or enhanced.

Microscopic examination is an extremely useful tool in the study and characterization of materials. Several important applications if microstructural examinations are as follow:

- To ensure that the associations between the properties and structure (and defects) are properly understood
- To predict the properties of materials once these relationships have been established
- To design alloys with new properly combinations
- To determine whether or not a material has been correctly heat treated
- To ascertain the mode of mechanical fracture.

2.3 Microscopic Techniques

Optical Microscopy

With optical microscopy, the light microscope is used to study the microstructure; optical and illumination systems are its basic elements. For materials that are opaque to visible light (all metals and many ceramics and polymers), only the surface is subject to observation and the light microscope must be used in a reflecting mode. Contrasts in the image produced result from differences in reflectivity of the various regions of the microstructure. Investigations of this type are often termed metallographic, since metals were first examined using this technique.

Normally, careful and meticulous surface preparations are necessary to reveal the important details of the microstructure. The specimen surface must first be ground and polished to a smooth and mirror like finish. This is accomplished by using successively finer abrasive papers and powders. The microstructure is revealed by a surface treatment using an appropriate chemical reagent in a procedure termed etching. The chemical reactivity of the grains of some single-phase materials depends on crystallographic orientation. Consequently, in a polycrystalline specimen, etching characteristics vary from grain to grain.

Also, small grooves form along grain boundaries as a consequence of etching. Since atoms along grain boundary regions are more chemically active, they dissolve at a greater rate than those within the grains. These grooves become discernible when viewed under a microscope because they reflect light at an angle different from that of the grains themselves.

When the microstructure of a two-phase alloy is to be examined, an etchant is often chosen that produces a different texture for each phase so that the different phases may be distinguished from each other.

Scanning Electron Microscopy

A more recent and extremely useful investigate tool is scanning electron microscope (SEM). The surface of a specimen to be examined is scanned with an electron beam and the reflected (or back-scattered) beam of electrons is collected, and then displayed at the same scanning rate on a cathode ray tube (similar to a CRT television screen). The image on the screen which may be photographed will represents the surface features of the specimen. The surface may or may not be polished and etched but it must be electrically conductive; a very thin metallic surface coating must be applied to nonconductive materials. Magnifications ranging from 10 to in excess of 50000 times are possible, as are also very great depth of field. Accessory equipment permits qualitative and semi quantitative analysis of the elemental composition of much localized surface area.

2.4 Finding the suitable Non-Destructive test for this project

Nondestructive testing (NDT) are noninvasive techniques to determine the integrity of a material, component or structure or quantitatively measure some characteristic of an object. In contrast to destructive testing, NDT is an assessment without doing harm, stress or destroying the test object. The destruction of the test object usually makes destructive testing more costly and it is also inappropriate in many circumstances.

X-ray diffraction (XRD) finds the geometry or shape of a molecule using X-rays. XRD techniques are based on the elastic scattering of X-rays from structures that have long range order. The most comprehensive description of scattering from crystals is given by the dynamical theory of diffraction

X-ray photoelectron spectroscopy (XPS) is a quantitative spectroscopic technique that measures the elemental composition, empirical formula, chemical state and electronic state of the elements that exist within a material. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the

kinetic energy (KE) and number of electrons that escape from the top 1 to 10 nm of the material being analyzed. XPS requires ultra high vacuum (UHV) conditions.

Energy dispersive X-ray spectroscopy (EDS) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It is one of the variants of XRF. As a type of spectroscopy, it relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing x-rays emitted by the matter in response to being hit with charged particles. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing x-rays that are characteristic of an element's atomic structure to be identified uniquely from one another.

2.5 How keris been made?

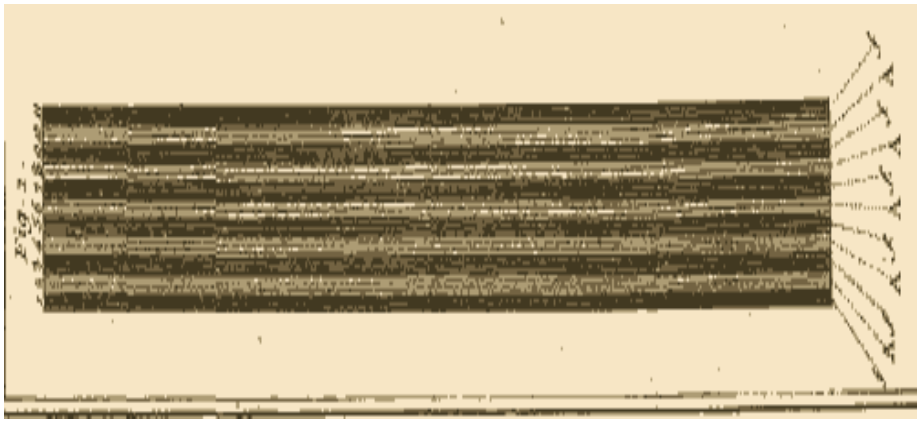


Figure 2: Art of Cutler; Layer of Iron and steel in making Damascus steel [2]

Start out by forging six thin plates of iron, exactly identical in all respects. Then forge five thin plates of steel, identical in form to those of iron, making in total eleven thin plates. Stack these plates one atop another, but be sure to put each steel plate between two of iron, which means starting and finishing with an iron plate. After that, grasp all the plates with a tongs and place this stack in a moderate fire. Raise the temperature so that all the plates heat uniformly, but do not allow any of them to burn.

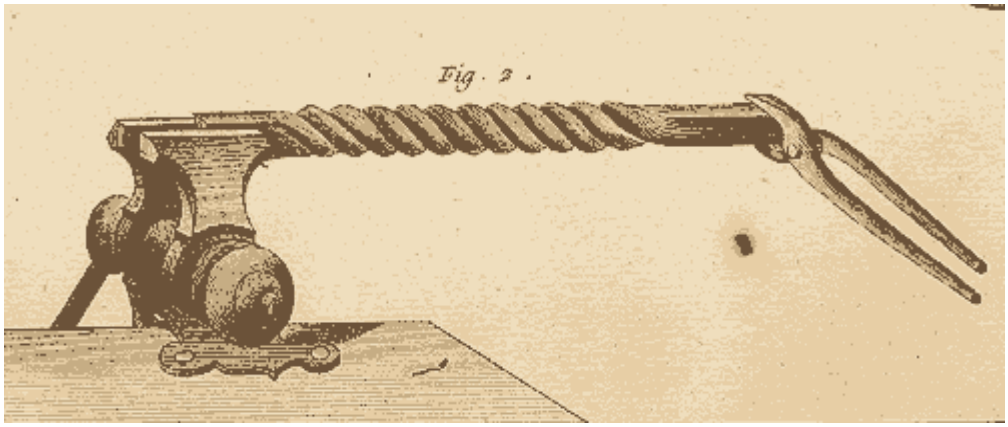


Figure 3: Art of Cutler; twisting process of the metal [3]

With stout tongs twist the material from one end to the other, as evenly as possible, so that it resembles a screw. This will form a strong tenacious covering, such that no effort or power can break it apart. The plates of soft iron are welded, married, and entwined with the ones of steel, forming together an extremely tough material, more tough than either component. Now, the iron and steel are well bonded together. Continue on the forging process until final shape of the keris blade is done.

2.6 What material usually used in making keris blade?

- ❖ Iron
    - Iron is a lustrous, ductile, malleable, silver-gray metal.
    - It is known to exist in four distinct crystalline forms.
    - Iron rusts in dump air, but not in dry air.
    - Iron is the most used of all the metals, including 95 % of all the metal tonnage produced worldwide.
- ❖ Carbon
    - There are several allotropes of carbon of which the best known are graphite, diamond , and amorphous carbon

- Diamond has a very low electrical conductivity while graphite is a very good conductor
- Under normal conditions, diamond has the highest thermal conductivity of all known materials.
- All allotropic forms are solids under normal conditions but graphite is the most thermodynamically stable
- All forms of carbon are highly stable, requiring high temperature to react even with oxygen

❖ Nickel

- Nickel is silvery-white, hard, malleable, and ductile metal.
- One of the iron group and it takes on a high polish.
- Good conductor of heat and electricity.
- The major use of nickel is in the preparation of alloys.
- Nickel alloys are characterized by strength, ductility, and resistance to corrosion and heat

2.7 What is Damascus Steel?

Bits of steel and iron and carbon are placed in a ceramic crucible and heated at very high temperatures until it all fused together. The high carbon content (usually above 2% - most steels used have less than 1% carbon) forms a lot of carbides, which precipitate out, forming lines on the surface of the steel. It is these lines of carbides that create the "watering" effect that damascus is so famous for.

What we call "damascus" today is really just pattern welded steel- smiths just weld together a few hundred layers of hard and soft steel and then etch in acid. The acid attacks the hard and soft steels at different rates resulting in a visible pattern on the steel.

2.8 Literature Review based on Mechanical Properties of Modern Fabricated Pattern Welded Damascus Steels

Introduction

There are two sorts of Damascus steels: "Genuine" or Wootz Damascus and Mechanical Pattern or Welded Damascus. In Russia the first one is named as Bulat (from Persian "Pulad"), while the second is named as Damascus steel simply.

Many of investigators have been occupied with Bulat/Wootz Damascus. In the middle of the last century the Russian metallurgist A. P. Anosov restored the technology of producing Bulat blades, and this production was glimmering at Zlatoust Arm Factory up to the end of 1919. Thorough works by Russian scientists N. I. Belaiew and N. T. Belaiew were published in the beginning of the century. In our days the blade smiths V. I. Basov (USSR) and A. H. Pendray (USA) were the first to make Bulat/Wootz Damascus blades. In his beautiful studies J. D. Verhoeven with co-workers laid the foundation for the theory of Wootz Damascus, for example.

Welded Damascus steel was fabricated and now is worked out in greater quantities than Wootz Damascus steel. However, in scientific respect the previous always was in the shadow of the latter. The author knows a couple of the papers on mechanical properties of genuine antique Bulat/Wootz Damascus blades. Of course, there are works devoted to the mechanical properties of modern fabricated Bulat steels. But mechanical characteristics of Welded Damascus steels are known worse.

The scope of the present work is to determine the mechanical properties of modern fabricated Welded Damascus steels from the best of Russian blade smiths.

Experimental

The samples were taken from works by blade smith V. D. Koptev (Tula, Russia) and "patriarch" of Russian blade smiths V. I. Basov (Suzdal', Russia). We had got the Basov's work in fully heat-treated state (quenched and tempered). The Koptev's work had been quenched only but not tempered. The load in microhardness test was 1 and 10 N. Because of heterogeneity and severe texture we restricted ourselves to the quality X-ray diffraction phase analysis. Yield strength, tensile strength, elongation and reduction of area had been determined by standard methods. Also, mean true strain $e_u = \ln (l_u / l_0)$ away necked region was been measured.

Result and Discussion

Phase analyses

Before the tempering the works had dual a - and g -phase sttructure. g -phase disappeared after 400 °C tempering. This is normal for carbon steels, where the temperature of full decomposition of retained austenite is about 300 °C.

Macrostructure

Microstructure of tested works is shown in Figure 4.

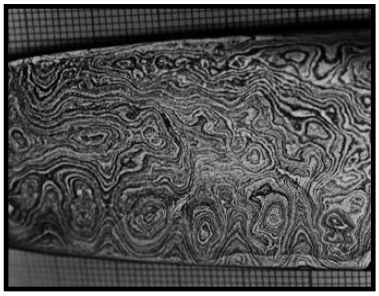
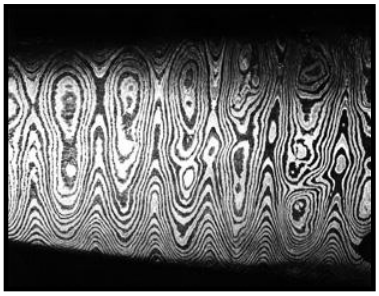
		
Koptev's work	Koptev's "Ni-damascus"	Macrostructure of the fragment from Basov’s work

Figure 4: Microstructure of tested works by Koptev and Basov [4]

Mechanical properties

Hardness

Very uneven hardness was observed in Damascus works. Even within a strand. Generally speaking, this is quite natural. As it is easy to see, there is no appreciable difference between hard strands of Koptev's work and 1.0% C steel. Behavior of the soft strands fit more to 0.35% C steel than to wrought iron, which usually used to fabricate Welded Damascus steel. Obviously, this was due diffusion carbon during fabrication.

Hardness of the zones in Basov's work differed considerably: at low hardness of strands, there are "spots" with high hardness (up to 6.3 Gpa after tempering at 400 °C). This is considerable more, than hardness of plan carbon steels after such tempering. The optical spectrometry confirmed absence of alloy elements in the "spots". Possibly, this is result of high forging strain hardening. But causes of that have not been ascertaining in details.

Conclusion

The samples of modern fabricated Welded Damascus steel (at list, the samples tested by us) do not exceed up-to-day high-carbon and low alloyed steels in combination hardness-toughness and they are considerably inferior to super steels.

In future, the author plans to fulfill the research of modern fabricated Bulat steels. Unfortunately, for the time being it is look like as impossible to investigate Bulat and Damascus by ancient fabrication. And the author will be very grateful to anybody who can help him with this.

2.9 Forging Process

Forging is the process by which metal is heated and is shaped by plastic deformation by suitably applying compressive force. Usually the compressive force is in the form of hammer blows using a power hammer or a press.

Forging refines the grain structure and improves physical properties of the metal. With proper design, the grain flow can be oriented in the direction of principal stresses encountered in actual use. Grain flow is the direction of the pattern that the crystals take during plastic deformation. Physical properties (such as strength, ductility and toughness) are much better in a forging than in the base metal, which has, crystals randomly oriented.

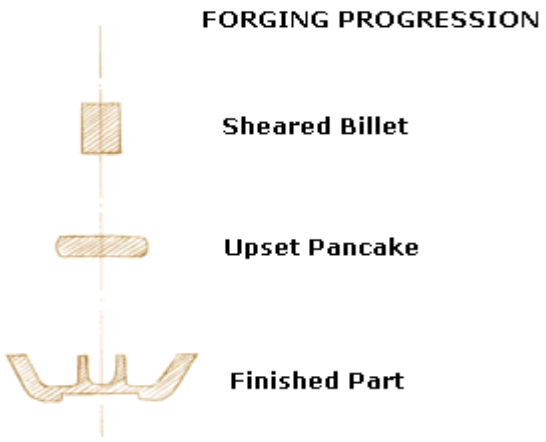


Figure 5: Forging Progression [5]

Forgings are consistent from piece to piece, without any of the porosity, voids, inclusions and other defects. Thus, finishing operations such as machining do not expose voids, because there aren't any. Also coating operations such as plating or painting are straightforward due to a good surface, which needs very little preparation.

Forgings yield parts that have high strength to weight ratio-thus are often used in the design of aircraft frame members.

A Forged metal can result in the following

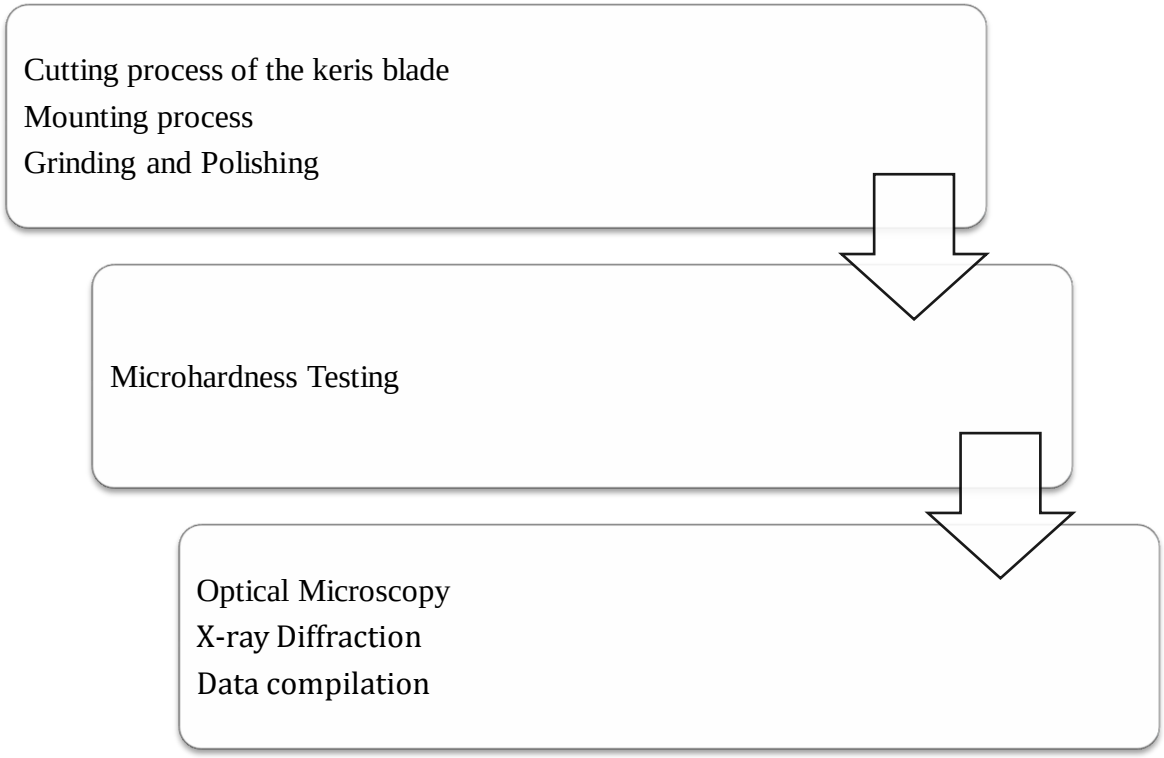
- Increase length, decrease cross-section, called drawing out the metal.
- Decrease length, increase cross-section, called upsetting the metal.
- Change length, change cross-section, by squeezing in closed impression dies. This results in favorable grain flow for strong parts

CHAPTER 3

METHODOLOGY

3.1 Project Work

Semester July 2010



Flow Chart 1: Project Flow for Final Year Project II Semester July 2010

3.2 Sample Preparation

Cutting Process

1. First, a keris blade need to be purchase and inspection on the keris blade surface need to be done
2. Only the keris with a Damascus pattern can be used for this project.
3. The keris blade will be cut into several part which will be used in the Optical Microscopy test, XRD and also Microhardness test.
4. The keris blade will be cut into certain size which is 1 cm x 1 cm.
5. According to the early calculation, there will be about 15 sample of the cubic keris blade will be achieved after the cutting process.
6. The cutting process first will use the milling machine. This step is to get the flat surface on the keris blade.
7. The surface of the keris blade will be removed for 3mm in order to get the inner surface of the keris blade.
8. Then, the keris blade will be cut into half using the abrasive cutter machine.
9. The lower part of the keris blade that been cut will be cut into small pieces of 10 in dimension of 1cm x 1cm and will be used later on in the mounting process.
10. The upper part of the keris blade will be cut using Electrical Discharge Machining (EDM) wirecut.

Figure 6: Cutting process using the milling machine

Figure 7: Cutting process to small part using Abrasive Cutter Machine

3.3 Optical Microscopy

Sample Preparation

For Metals

When preparing samples for microscopy, it is important to produce something that is representative of the whole specimen. It is not always possible to achieve this with a single sample. Indeed, it is always good practice to mount samples from a material under study in more than one orientation. The variation in material properties will affect how the preparation should be handled, for example very soft or ductile materials may be difficult to polish mechanically.

Mounting

Mounting of specimens is usually necessary to allow them to be handled easily. It also minimizes the amount of damage likely to be caused to the specimen itself.

The mounting material used should not influence the specimen as a result of chemical reaction or mechanical stresses. Specimens can be hot mounted (at around 200 °C) using a mounting press, either in a thermosetting plastic (*e.g.* phenolic resin), or a thermo softening plastic (*e.g.* acrylic resin). If hot mounting will alter the structure of the specimen a cold-setting resin can be used, *e.g.* epoxy, acrylic or polyester resin. Porous materials must be impregnated by resin before mounting or polishing, to prevent grit, polishing media or etchant being trapped in the pores, and to preserve the open structure of the material.

A mounted specimen usually has a thickness of about half its diameter, to prevent rocking during grinding and polishing. The edges of the mounted specimen should be rounded to minimise the damage to grinding and polishing discs.

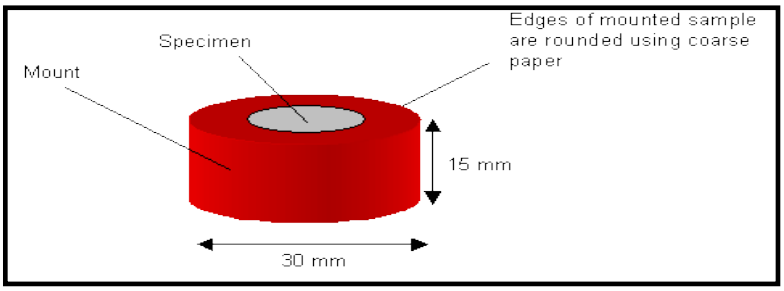


Figure 8: A diagram of a mounted specimen [8]

Figure 9: Auto Mounting Press Machine

Grinding

Surface layers damaged by cutting must be removed by grinding. Mounted specimens are ground with rotating discs of abrasive paper flushed with a suitable coolant to remove debris and heat, for example wet silicon carbide paper. The coarseness of the paper is indicated by a number: the number of grains of silicon carbide per square inch. So, for example, 180 grit paper is coarser than 1200.

The grinding procedure involves several stages, using a finer paper (higher number) for each successive stage. Each grinding stage removes the scratches from the previous coarser paper. This is more easily achieved by orienting the specimen perpendicular to the previous scratches, and watching for these previously oriented scratches to be obliterated. Between each grade the specimen is washed thoroughly with soapy water to prevent contamination from coarser grit present on the specimen surface. Typically, the finest grade of paper used is the 1200, and once the only scratches left on the specimen are from this grade, the specimen is thoroughly washed with water, followed by alcohol and then allowed to dry.

It is possible to determine the start point for grinding using the following empirical relationship where the width of the largest scratch is measured under a microscope:

$$\text{Paper grid size} = \frac{16000}{Width\ of\ largest\ \ scratch\ (in\ microns\ )}$$

This prevents putting more damage into the sample than already exists; the coarsest grades of paper are often not useful.

Figure 10: The grinding process

Polishing

Polishing discs are covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant. Particles of two different grades are used : a coarser polish - typically with diamond particles 6 microns in diameter which should remove the scratches produced from the finest grinding stage, and a finer polish – typically with diamond particles 1 micron in diameter, to produce a smooth surface. Before using a finer polishing wheel the specimen should be washed thoroughly with warm soapy water followed by alcohol to prevent contamination of the disc. Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen, if the specimen is particularly susceptible to mechanical damage (or excessive force is used in the grinding and polishing stages) debris can become embedded in the surface and plastic deformation may exist below the surface. Electro polishing or chemical polishing can be used to remove this, leaving an undisturbed surface.

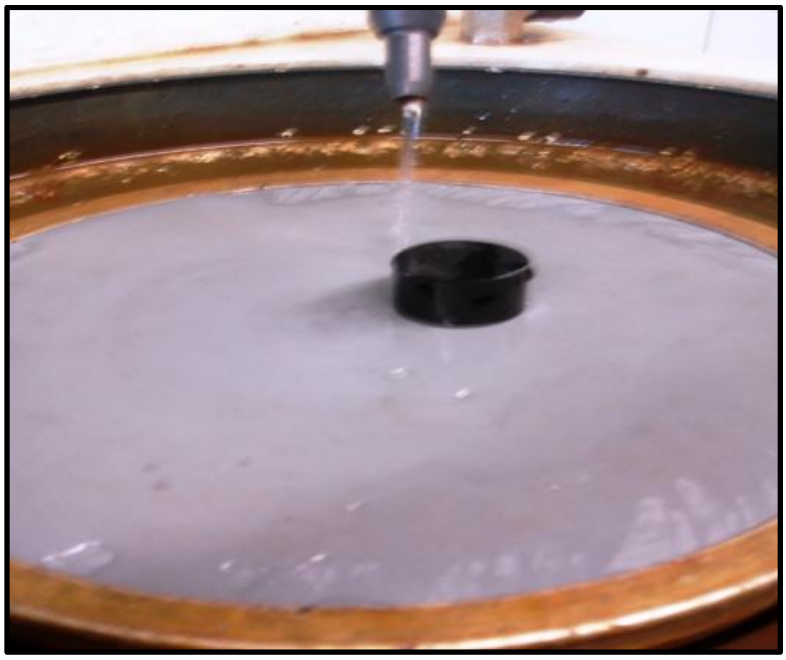


Figure 11: The polishing process

Etching

Etching is used to reveal the microstructure of the metal through selective chemical attack. It also removes the thin, highly deformed layer introduced during grinding and polishing.

In alloys with more than one phase, etching creates contrast between different regions through differences in topography or reflectivity. The rate of etching is affected by crystallographic orientation, the phase present and the stability of the region. This means contrast may arise through different mechanisms – therefore revealing different features of the sample.

In all samples, etchants will preferentially attack high energy sites, such as boundaries and defects.

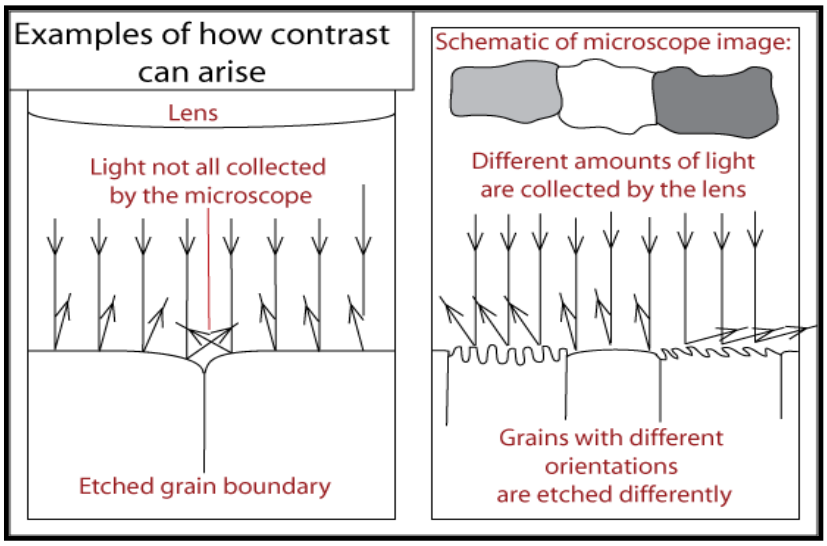


Figure 12: Example of how contrast can arise [9]

The specimen is etched using a reagent. For example, for etching stainless steel or copper and its alloys, a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid is used. This is applied using a cotton bud wiped over the surface a few times (Care should be taken not to over-etch - this is difficult to determine, however, the photos below may be of some help). The specimen should then immediately be washed in alcohol and dried.

Following the etching process there may be numerous small pits present on the surface. These are etching pits caused by localized chemical attack and, in most cases, they do not represent features of the microstructure. They may occur preferentially in regions of high local disorder, for example where there is a high concentration of dislocation.

If the specimen is over etched, such as etched for too long, these pits tend to grow, and obscure the main features to be observed. If this occurs it may be better to grind away the poorly etched surface and re-polish and etch, although it is important to remember what features you are trying to observe – repeatedly grinding a very thin sample may leave nothing to see.

Ideally the surface to be examined optically should be flat and level. If it is not, the image will pass in and out of focus as the viewing area is moved across the surface. In

addition, it will make it difficult to have the whole of the field of view in focus - while the centre is focused, the sides will be out of focus. By using a specimen leveling press (shown below) this problem can be avoided, as it presses the mounted specimen into plasticize on a microscope slide, making it level. A small piece of paper or cloth covers the surface of the specimen to avoid scratching.

3.4 Operational Procedure for X-ray Diffraction machine

Safety Precaution:

Due to the danger of possible exposure to stray x-ray radiation, extra care should be taken all the times. Lead body protection should be worn while the x-ray machine is activated and the room should be vacant during film exposure.

Procedure:

1. Mount the crystal sample on the goniometer (either wax or glue are typically used). Note that the locking 'key' may be used in the goniometer in order to prevent the crystal from rotation about the x-ray once mounted to the goniometer.
2. Turn on the cooling water. First, open the outlet valve (the one with the flow meter), and then open the inlet valve.
3. Plug the x-ray machine into the wall socket (push plug in, then turn clockwise until tight). Check that both the voltage and current control knobs are turned off (all the way counter-clockwise) and the aperture on the x-ray port is closed (pushed in).
4. Turn the activation key to the on position, press the 'start' button and then press the 'x-ray on' button.
5. Let the machine warm up for approximately 15 to 20 minutes.
6. Place the 5 cm spacer on the mounting rail. Next, place the goniometer on the rail flush with the 5 cm spacer. Check that they are pushed tight against the x-ray port, and then clamp them into position.

7. Align the surface of the crystal using the eyepiece. The front surface of the crystal should be centered in the view from the eyepiece when the goniometer is set to (0, 0).
8. Adjust the crystal orientation to the desired angle.
9. Check that the exposures switch is down and that the loading lever is in the 'load' position. Wait until film is warmed to room temperature before proceeding (use Polaroid '57' professional high speed between 4 x 5 instant sheet films).
10. Insert film into carriage with the '57' label facing toward the crystal. The film should be pushed down into the carriage until it engages with a 'click' and then pulled upward until resistance is felt.
11. Push exposure switch up.
12. Increase the voltage to 20 to 30 kV and the current to 15 to 20 mA.
13. During the next two steps x-ray will be bombarding the crystal. Due to the danger of stray x-rays, protection should be worn and it is best to leave the room during the film exposure process.
14. Open the x-ray aperture by pulling the metal slide on the x-ray port outward. Warning: there is now x-ray emanating from the x-ray port. Allow 3 to 5 minutes for the film to be exposed then close x-ray aperture by pushing metal slide inward. Decrease the current and voltage back to 0.
15. Push the exposure switch down.
16. Push the film all the way down into the carriage and then flip the loading lever to the 'process' position.
17. Smoothly pull the film out of the carriage in one quick motion. Wait 15 to 20 seconds for film to develop then remove the front paper cover from the film (avoid contact with the developing chemicals). If desired, apply the 'print coater' to the picture surface when dried.
18. The relation between distance on the picture and the angle of the goniometer is given by the following equation (for the 5 cm spacer): Angle = ½ x arctan (distance / 5 cm). Draw axis on the picture that passes through the 4 dark dots (near the center) that correspond to the relative orientation of the film and x-ray source. Draw axis through the bright symmetry lines due to diffraction from the

crystal. Measure the Cartesian distances between the two origins and use the preceding formula to calculate the two corresponding angles (in the x and y planes). Rotate the goniometer by the calculated angles in the direction toward the origin of the symmetry axis.

19. If the locking 'key' was not used in the goniometer mount, the crystal may be rotated so that the symmetry axis is parallel to the x-ray (and goniometer) axis.
20. Repeat steps 7 to 19 if necessary. The voltage, current, exposure time and developing time may all be varied in order to optimize the results.
21. Once finished, press the 'x-ray off' button and wait several minutes.
22. Press the 'stop' button, turn the key to the off position and then remove the plug from the wall socket.
23. Close the cooling water inlet valve and the close the outlet valve.

3.5 Microhardness Testing

The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf. The full load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation.

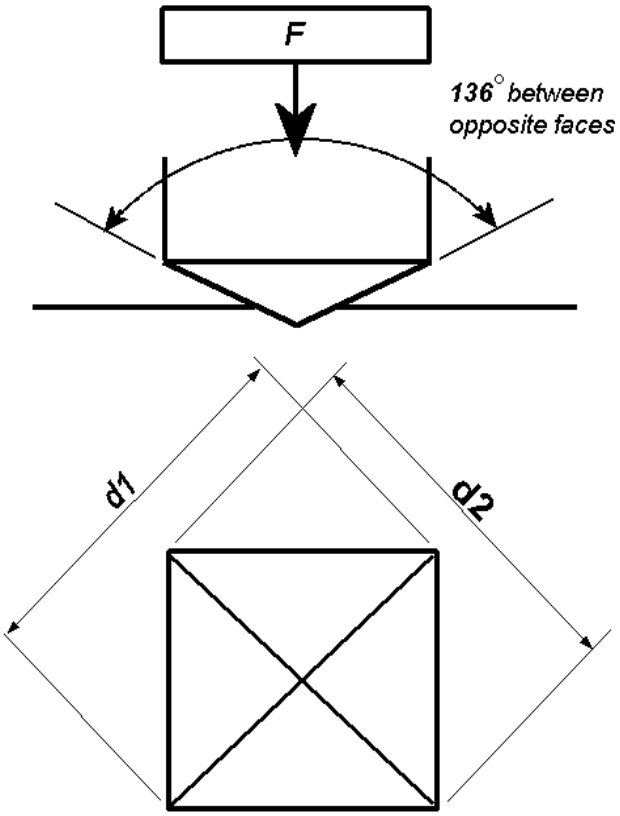


Figure 13: Indentation on the specimen

F= Load in kgf

d = Arithmetic mean of the two diagonals, *d1* and *d2* in mm

HV = Vickers hardness

$$HV = \frac{2F\sin\frac{136^o}{2}}{d^2} \qquad HV = 1.854\frac{F}{d^2} \text{ approximately}$$

When the mean diagonal of the indentation has been determined the Vickers hardness may be calculated from the formula, but is more convenient to use conversion tables. The Vickers hardness should be reported like 800 HV/10, which means a Vickers hardness of 800, was obtained using a 10 kgf force. Several different loading settings give practically identical hardness numbers on uniform material, which is much better

than the arbitrary changing of scale with the other hardness testing methods. The advantages of the Vickers hardness test are that extremely accurate readings can be taken, and just one type of indenter is used for all types of metals and surface treatments. Although thoroughly adaptable and very precise for testing the softest and hardest of materials, under varying loads, the Vickers machine is a floor standing unit that is more expensive than the Brinell or Rockwell machines.

3.6 Tools and Machine Required

3.6.1 Optical Microscopy

- To study the metallographic of the keris blade
- To study on the microstructure and optical texture of the keris blade

3.6.2 X-ray Diffraction

- To study on the crystallographic phases of the keris blade
- To confirm whether the element is exists in the keris blade.

3.6.3 Microhardness Testing

- To determine the hardness of the keris blade at the three axis of the keris blade.

3.7 Expected Result

Figure 14

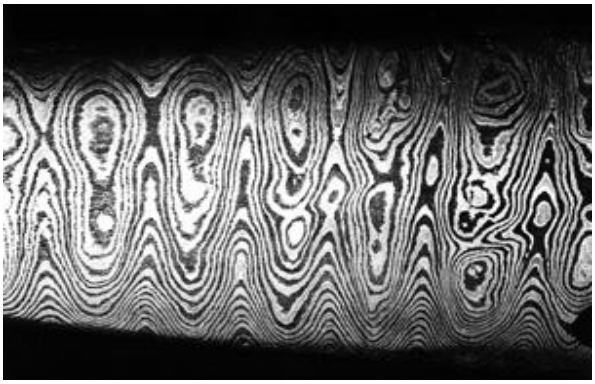


Figure 15

Figure 16

Figure 14, 15 and 16 shows the microstructure of the Damascus steel [4]

CHAPTER 4

RESULT AND DISCUSSION

4.1 Sample Preparation Process

Sample preparation need to be done on both keris from Kuala Kangsar and Perlis. Keris from Kuala Kangsar will be noted as Sample 1 while keris from Perlis will be noted as Sample 2. Sample 1 is tempered and quenched while Sample 2 is quenched only.

Cutting a specimen

It important to know that preparation of a specimen may change the microstructure of the material. For example, it may change through heating, chemical attack, or mechanical damage. The amount of damage depends on the method by which the specimen is cut and the material itself.

Cutting with abrasives may cause a large amount of damage, whilst the use of a low-speed diamond saw can cause fewer problems. There are many different cutting methods, although some are used only for specific specimen types.

Mounting Process

The mounting process will consist of the specimens from the 3-axis; x-axis, y-axis and z-axis. The heat time required is set to 8 minutes and the cool time is 5 minutes. The pressure used in the mounting process is 4000 psi.

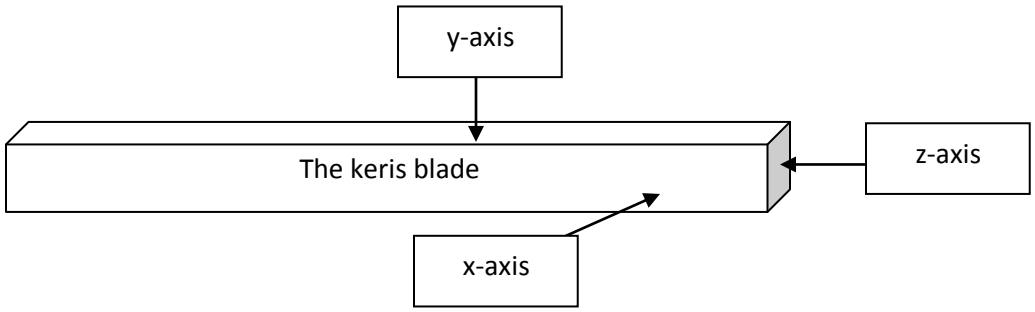


Figure 17: The axis where the sample will be taken for the mounting process

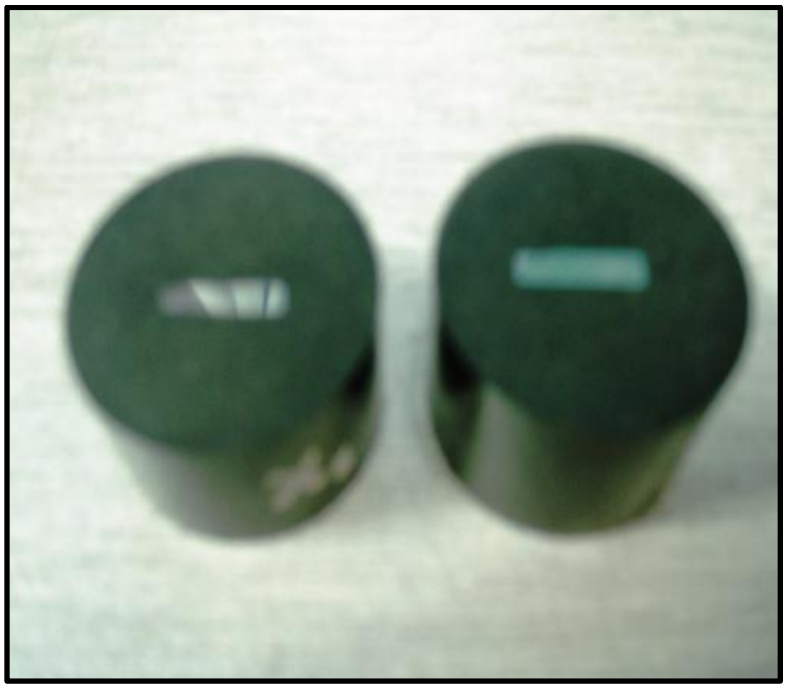


Figure 18: x-axis of Sample 1 and Sample 2

Figure 19: y-axis of Sample 1 and Sample 2

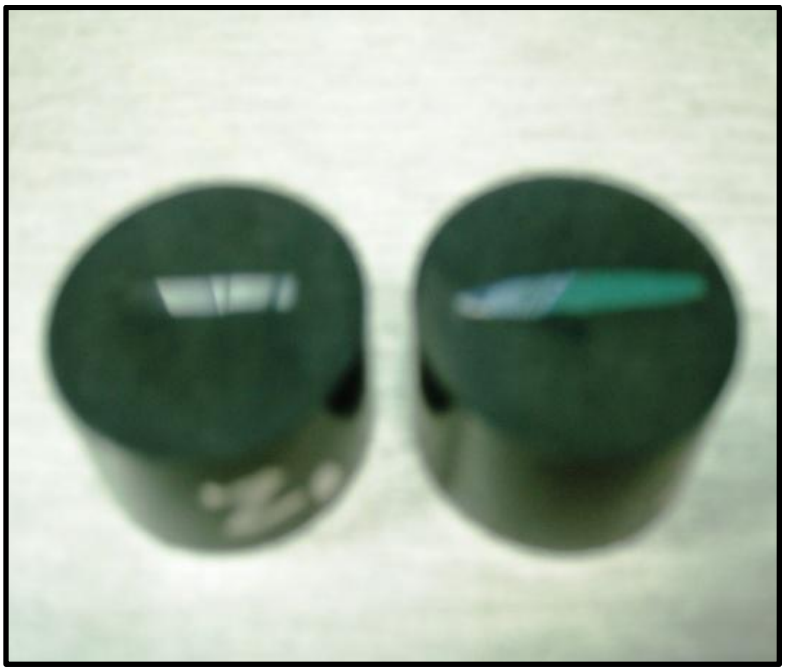


Figure 20: z-axis of Sample 1 and Sample 2

Grinding Process

The grinding process will be start with the less fine of the paper grade. First, place the sand paper on the grinder. Then, open the grinder spinner and the water. Place the mount specimen on the grinder slowly to avoid depth cut through the sample. Repeat the process with different paper grade starting with the less fine paper grade. The paper grade that been used are arrange in the table below:

Process	Paper Grade
1	240/P280
2	400/P800
3	600/P1200
4	P2400
5	P4000

Table 1: Paper Grade Value for each Process

Etching Process

The purpose of etching is to optically enhance microstructure features such as grain size and phase features. Etching selectively alters these microstructure features based on composition, stress or crystal structure. The most common technique for etching is selective chemical etching and numerous formulations have been used over the years. Both Sample 1 and Sample 2 are made from carbon steel. So, the best etchant for carbon steel is as below.

Etchant for Carbon Steel:

1. NITAL (2%, 15%)

HNO_3 (Nitric Acid) + Ethanol / Methanol

Purpose: will reveals alpha grain boundaries and constituents

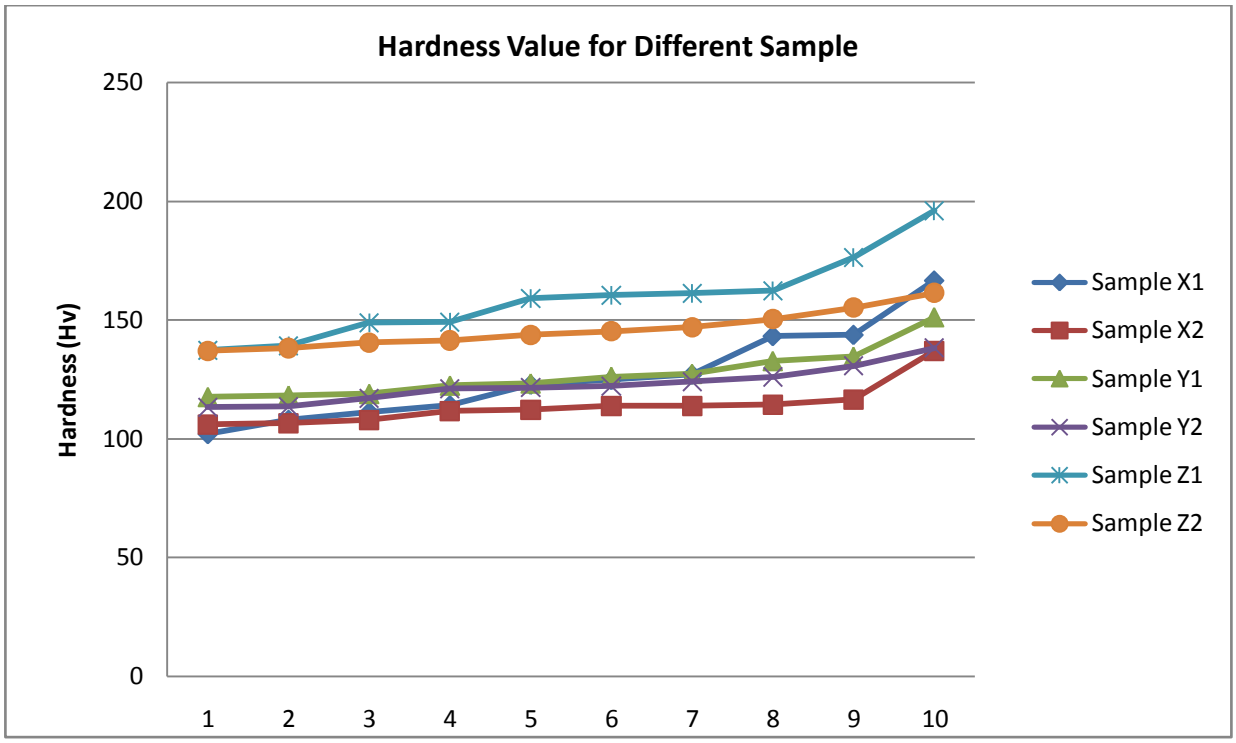
4.2 Microhardness Testing

The result of microhardness testing is showed in the table below:

Sample Number	Hardness Value (Hv)									
Sample X1	102	108	111.2	114.1	122.8	124.8	127	143.3	143.8	166.7
Sample X2	106.1	106.7	108	111.7	112.4	113.8	114	114.4	116.5	137.1
Sample Y1	117.7	118.3	119	122.5	123.4	126.2	127.4	132.8	134.6	151.2
Sample Y2	113.3	113.6	117.1	121.1	121.5	122.2	124.1	126	130.7	138.2
Sample Z1	137.3	139.1	148.9	149.2	159.1	160.5	161.2	162.4	176.3	196.1
Sample Z2	137.1	138.3	140.6	141.5	143.8	145.3	147	150.4	155.2	161.4

Table 2: Sample number with the hardness value from 10 different place on the sample

From the table, hardness value for different sample is plotted to give more clear view on the hardness value.



Graph 1: Hardness value for different sample

From the graph, the hardness value for both sample X_1 and sample X_2 is lower than other sample. This is due to the mechanical damage of the properties during the cutting process. Cutting with abrasives may cause a large amount of damage and weaken the hardness of the keris blade. From the 50x magnification of x-axis microstructure, it appears that lot of austenite phase still occurs in the sample compare to martensite phase. This mean that the hardness of x-axis is lower since austenite phase is less hard than the martensite phase.

The hardness value of sample Y_1 and Y_2 also low as sample Y represent the surface of the keris blade. In earlier process, in order to get the flat surface and remove the outer layer of the keris blade, milling process been used on the keris blade. The cutting fluid did not apply during this process and this may damage the microstructure and properties of the keris blade. The heat that collected during the milling process is the main cause of this problem. From the 50x magnification of y-axis microstructure, it shows that

austenite phase is still occurring in the sample. This means that y-axis also less in the hardness.

The graph shows that sample Z_1 and sample Z_2 have the highest hardness value from the other two samples from x-axis and y-axis. During the forging process, the blade-smith is concentrating on the z-axis strength as z-axis is frequently used for keris in action such as stabbing. So, the hardness of the keris should be the highest in z-axis. From the microstructure picture, it shows that z-axis fully achieve the martensite phase. This will result in high hardness in the z-axis.

4.3 Optical Microscopy Result

Result for first run of Optical Microscopy Testing (without etching)

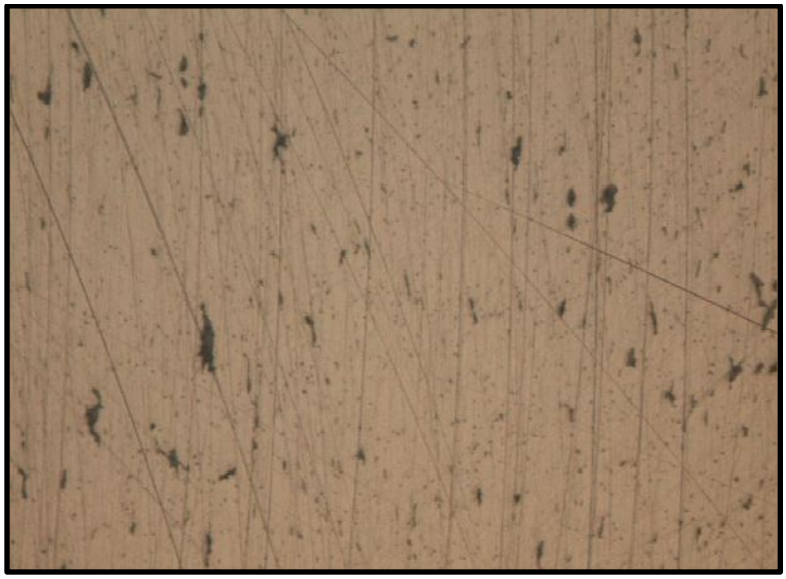


Figure 21: Optical Microscopy image for x-axis

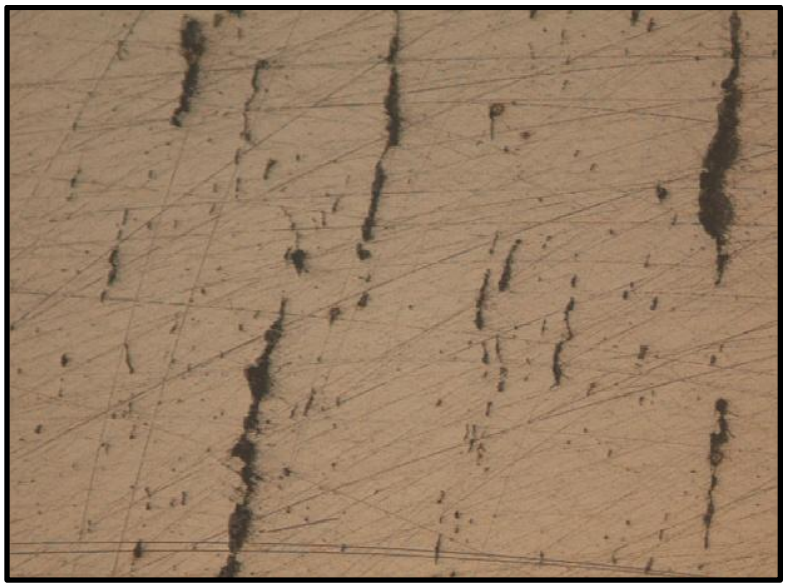


Figure 22: Optical Microscopy image for y-axis

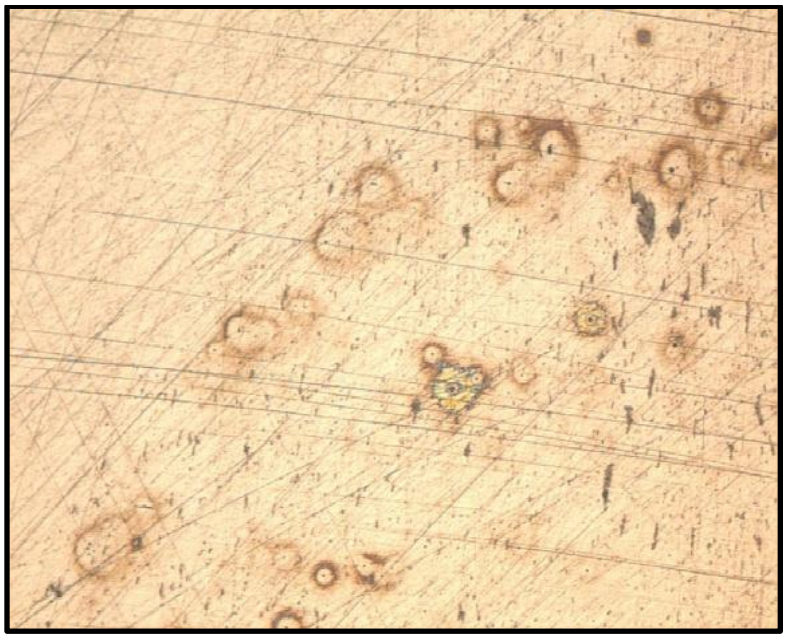


Figure 23: Optical Microscopy image for z-axis

The image of optical microscopy scanning from the three axes (Figure 20, 21 and 22) is not good and clear as there are still too many scratches on the surface of the sample. This is due to the less fine grade of sand paper been used during the grinding process. Grinding and polishing process need to be done back to get a better surface finish on the sample. Etching process also needs to be done on the sample so that good image of the

Damascus pattern can be seen from the optical microscopy result. The time of exposure of acid and the sample surface also need to be optimized to get better image result.

Result for second run of Optical Microscopy Testing (with etching)

Etching process need to be done in order to get a better result for the Optical Microscopy testing. First, we need to determine the etching solution that will be used on the sample surface. Since, the sample is low carbon steel, the best etching solution should be the Nital acid. Wash the sample surface with ethanol and rinse it with water. Dry the surface and exposed the surface of the sample to the Nital Acid about five to ten seconds. Do not let the Nital acid dry because it will burn the surface of the sample. Then, rinse the surface with water and dry it up. Now, the sample is ready for Optical Microscopy test.

Optical Microscopy Image for Sample 1 x-axis

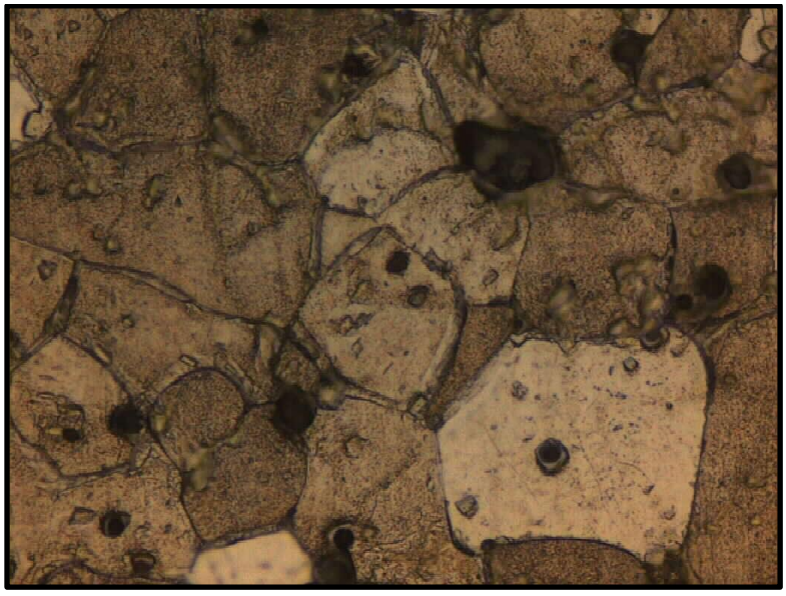


Figure 24: 500x Magnification on Sample 1 x-axis

Figure 25: 1000x Magnification on Sample 1 x-axis

Figure 26: 1500x Magnification on Sample 1 x-axis

Optical Microscopy Image for Sample 1 y-axis

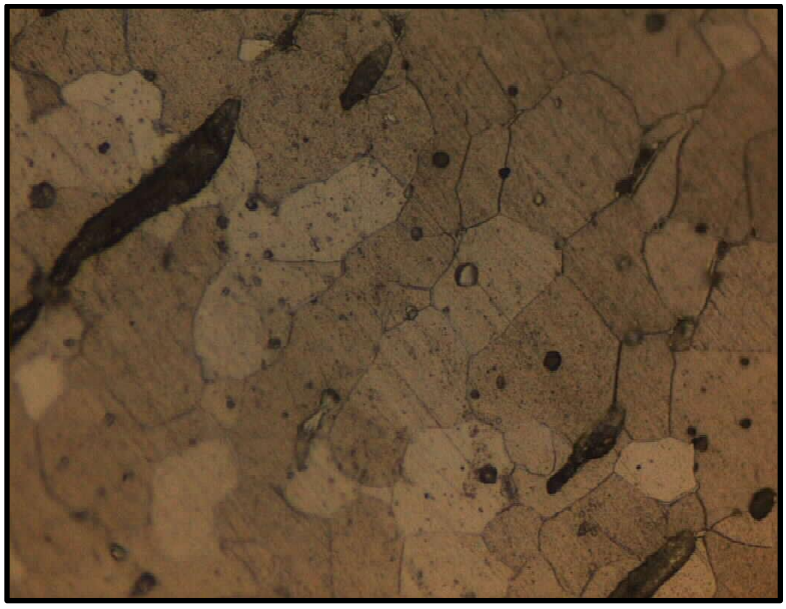


Figure 27: 500x Magnification on Sample 1 y-axis

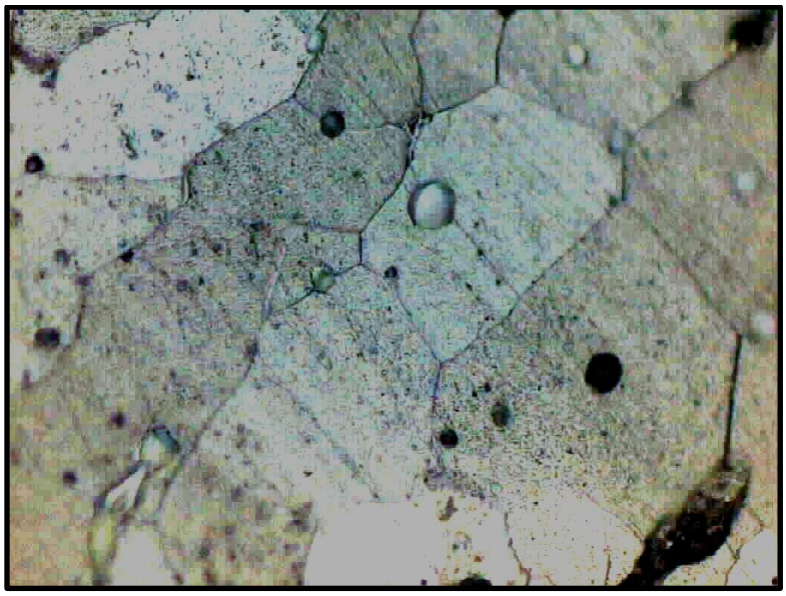


Figure 28: 1000x Magnification on Sample 1 y-axis

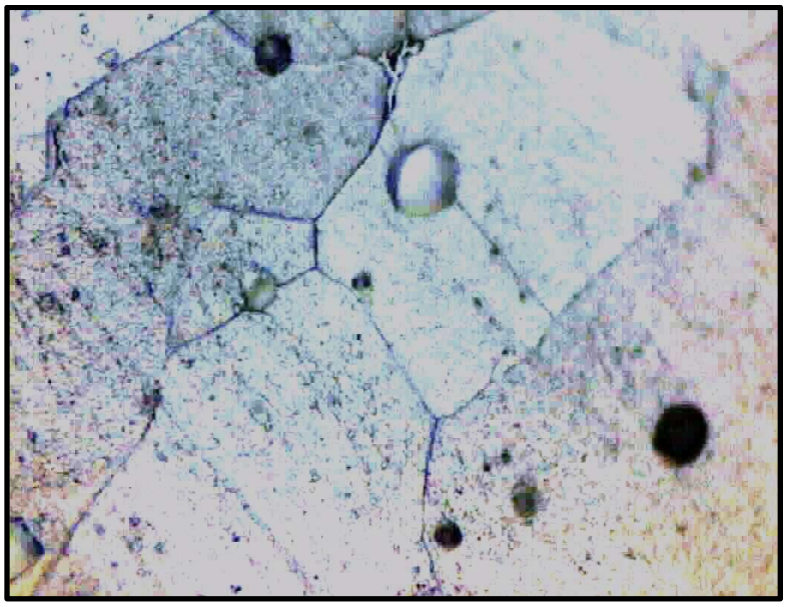


Figure 29: 1500x Magnification on Sample 1 y-axis

Optical Microscopy Image for Sample 1 z-axis

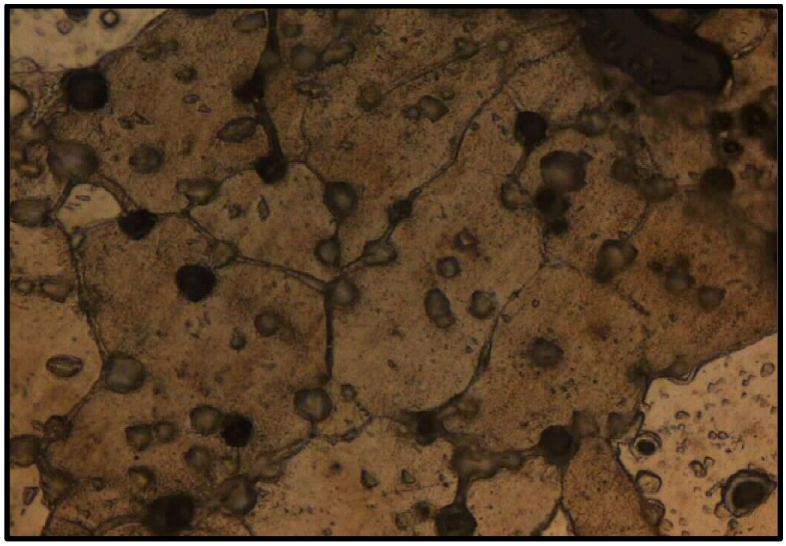


Figure 30: 500x Magnification on Sample 1 z-axis

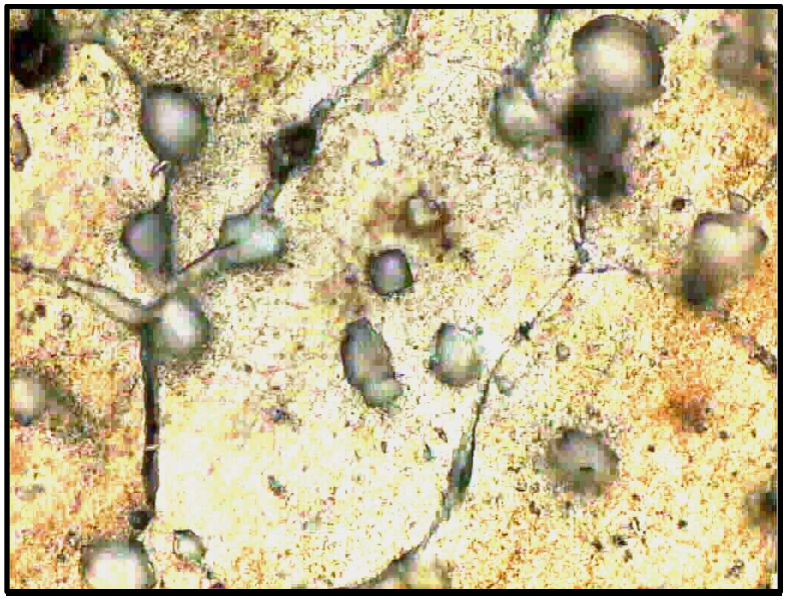


Figure 31: 1000x Magnification on Sample 1 z-axis

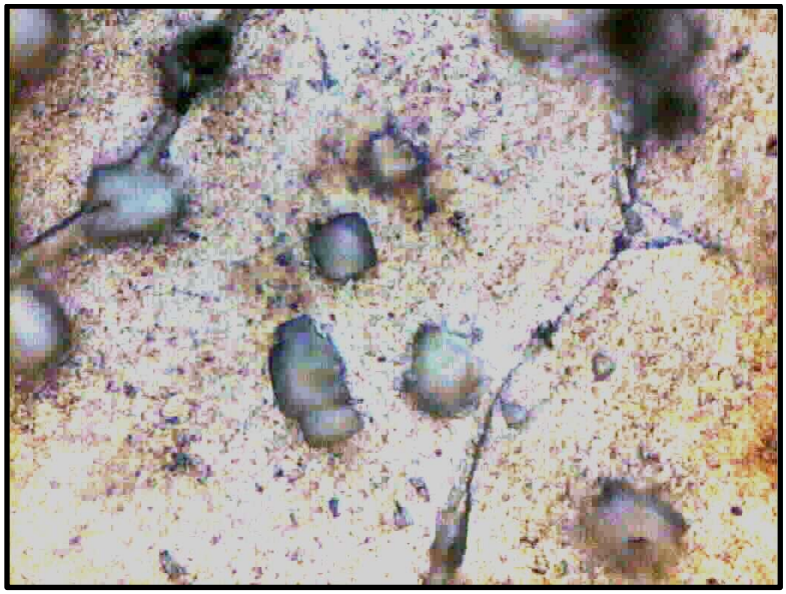


Figure 32: 1500x Magnification on Sample 1 z-axis

Optical Microscopy Image for Sample 2 x-axis

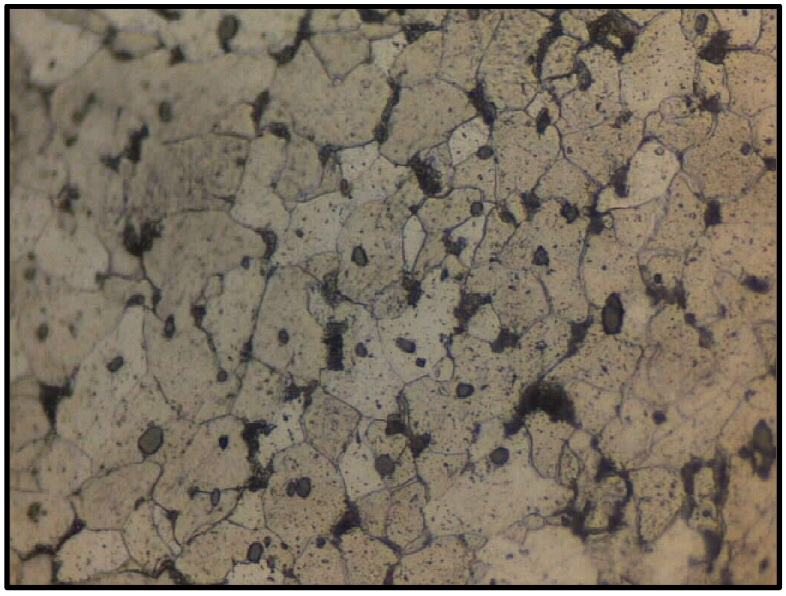


Figure 33: 500x Magnification on Sample 2 x-axis

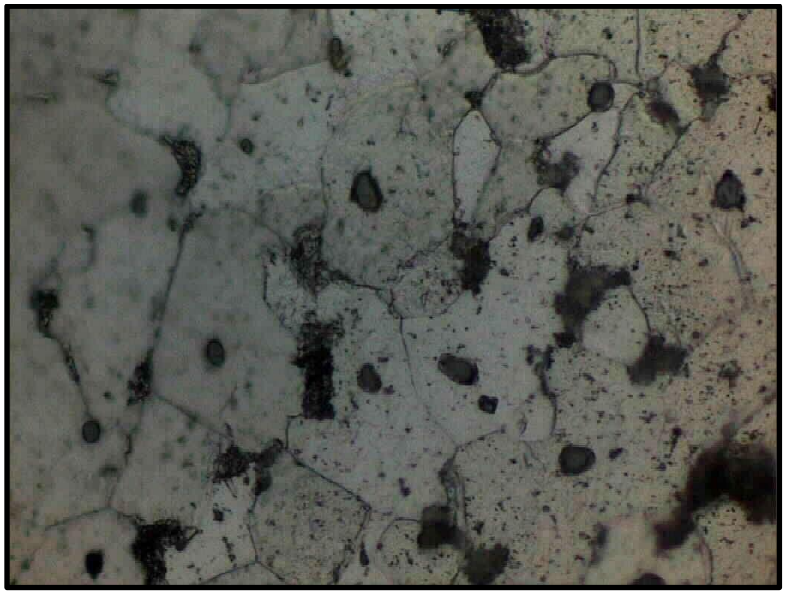


Figure 34: 1000x Magnification on Sample 2 x-axis

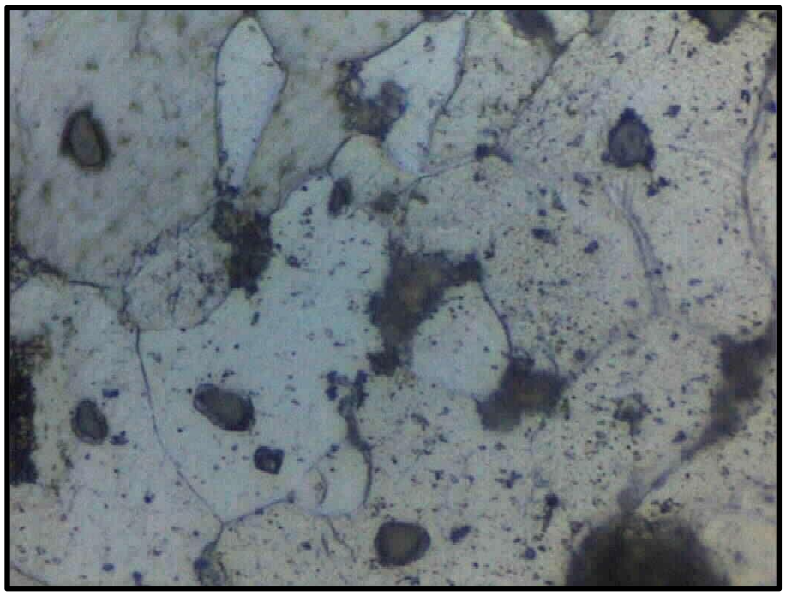


Figure 35: 1500x Magnification on Sample 2 x-axis

Optical Microscopy Image for Sample 2 y-axis

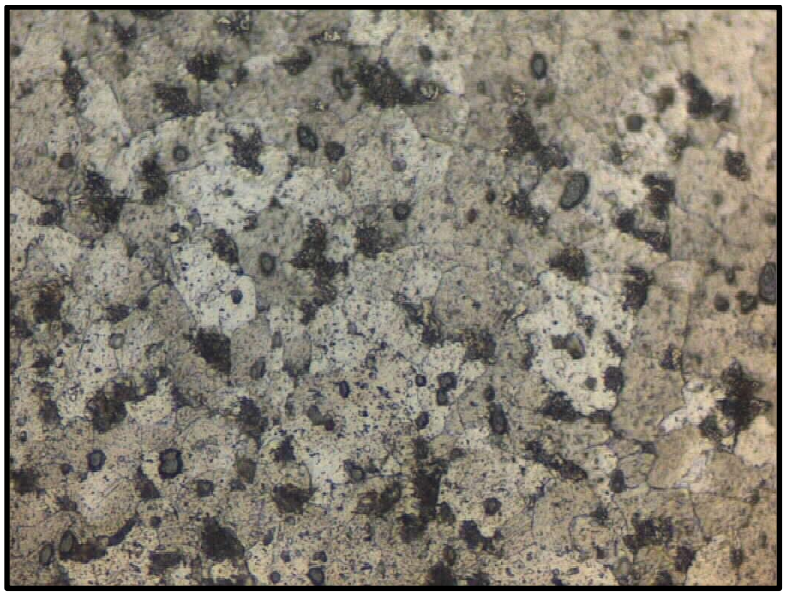


Figure 36: 500x Magnification on Sample 2 y-axis

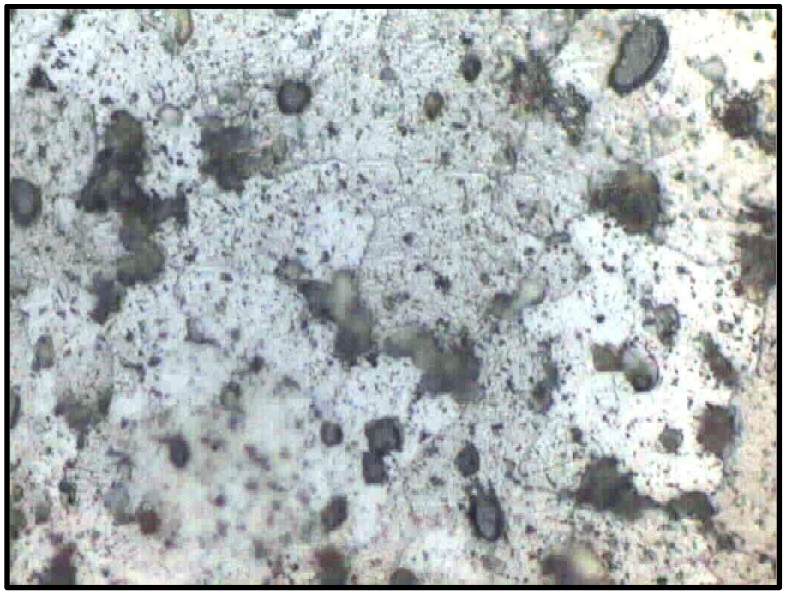


Figure 37: 1000x Magnification on Sample 2 y-axis

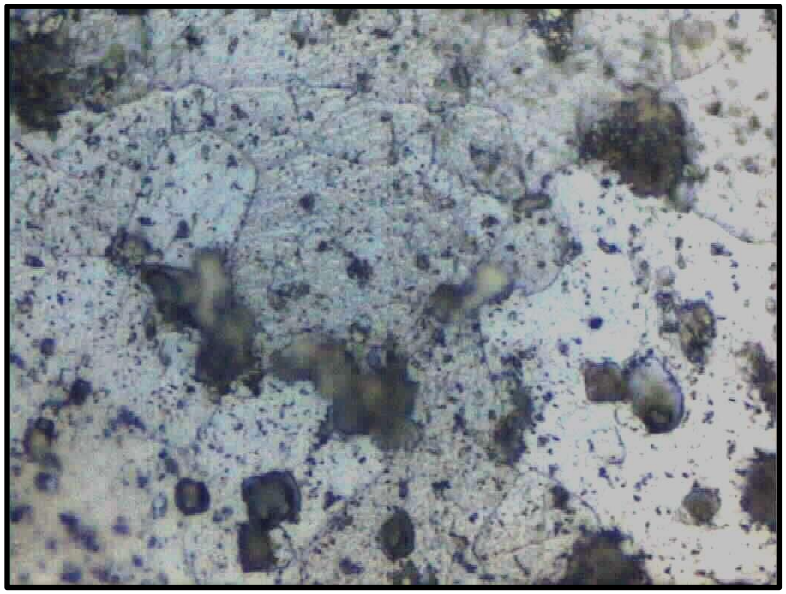


Figure 38: 1500x Magnification on Sample 2 y-axis

Optical Microscopy Image for Sample 2 z-axis

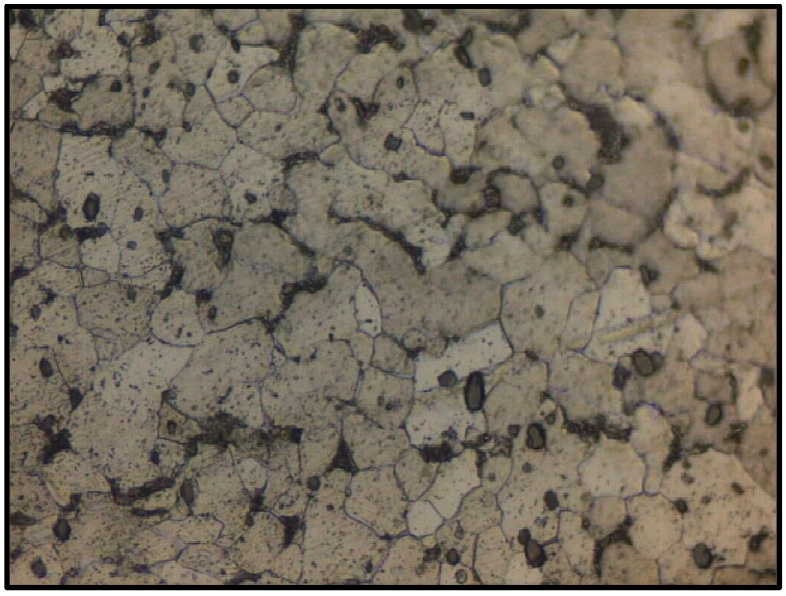


Figure 39: 500x Magnification on Sample 2 z-axis

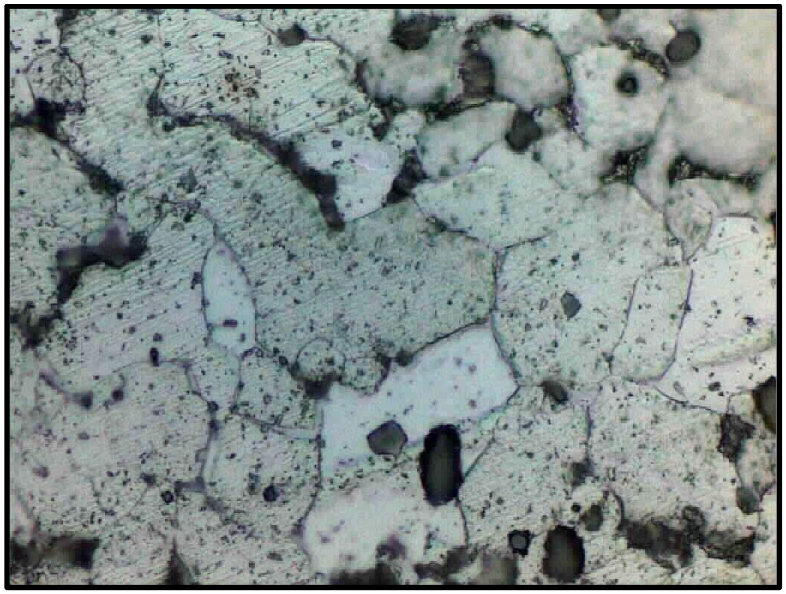


Figure 40: 1000x Magnification on Sample 2 z-axis

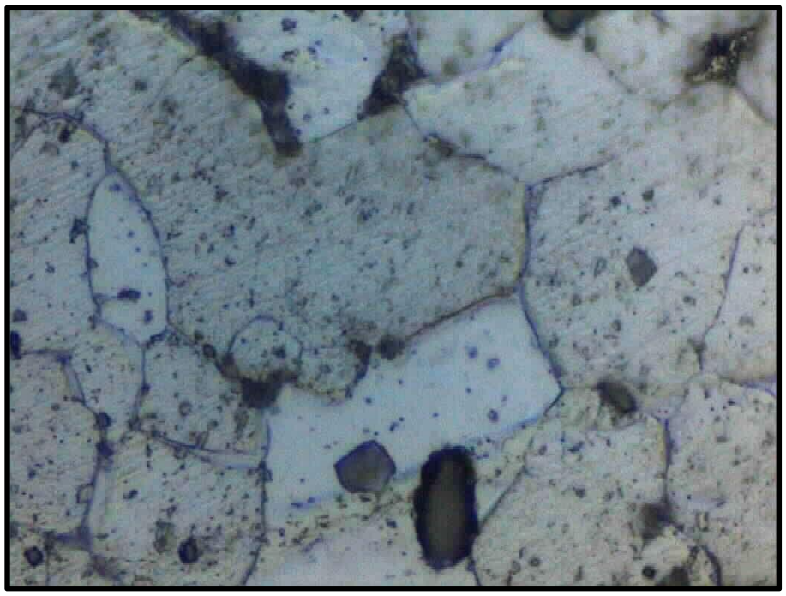


Figure 41: 1500x Magnification on Sample 2 z-axis

For Optical Microscopy, we observe the microstructure of keris blade from two different keris maker which label as Sample 1(Kuala Kangsar) and Sample 2(Perlis). Sample 1 is tempered and quenched while Sample 2 is quenched only. From the graph, the z-axis for both sample 1 and sample 2 shows the highest value of the hardness. This is due to many layer of metal form during the forging process. For sample 2, the hardness aspect may possibly result from high forging strain hardening. For sample 1, the hardness aspect is due to diffusion carbon during fabrication. At glowing state, on the atomic level, carbon atoms are on the move. They are much smaller than iron atoms and can travel in between them to move via a process called “diffusion”. For both sample 1 and 2, there are austenite phases which is due to the quenched process. Martensite phase will affect the hardness of both samples. Cutting with abrasive during the cutting process may damage the hardness and microstructure of both samples. At forging or quenching heat the iron atoms arrange themselves in such a way that allows many more pathways for the carbon to travel, so it is readily dissolvable in iron at these temperatures much more so than at room temperature. Austenite is a solid solution of this alloying element in face centered cubic iron which is confirmed by XRD results. Austenite phase allow keris maker to forge the metal and it is the phase that readily exist at high temperature and

responsible in most of heat treating and forging operation. Retained austenite is the white patches between needles in a carbon crucible steel. The surrounding dark area is the fine pearlite.

4.4 X-ray Diffraction Result

Result of X-ray Diffraction for Sample 1:

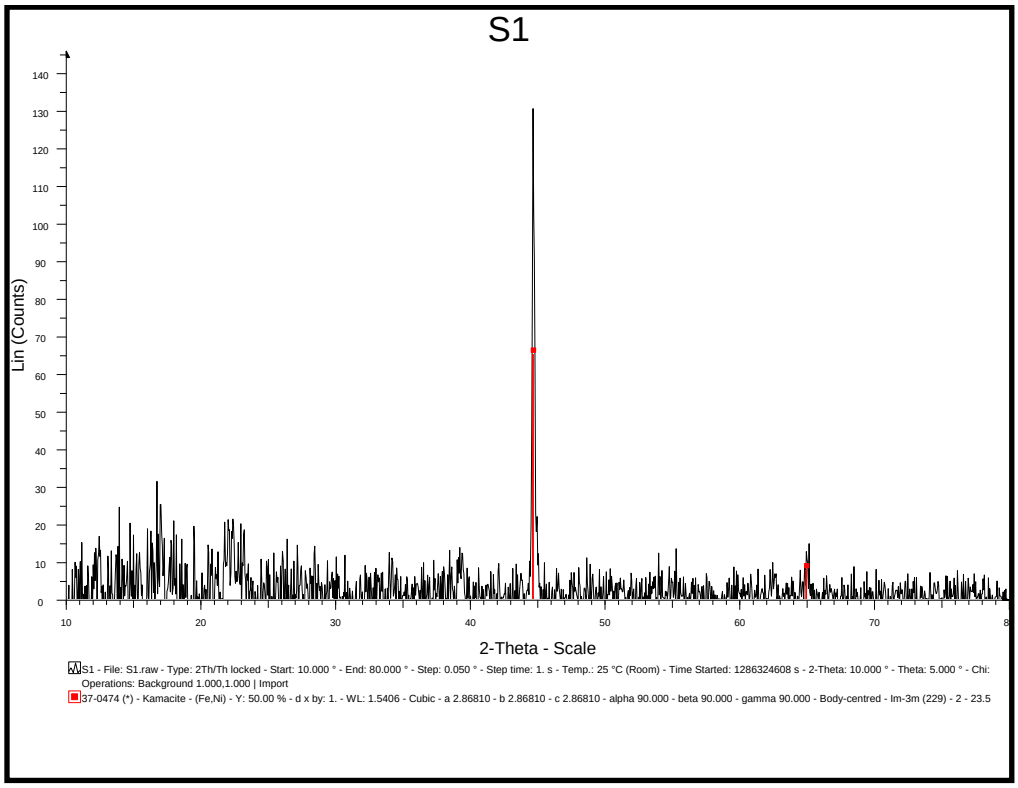


Figure 42: EVA Phase Identification Result of Sample 1

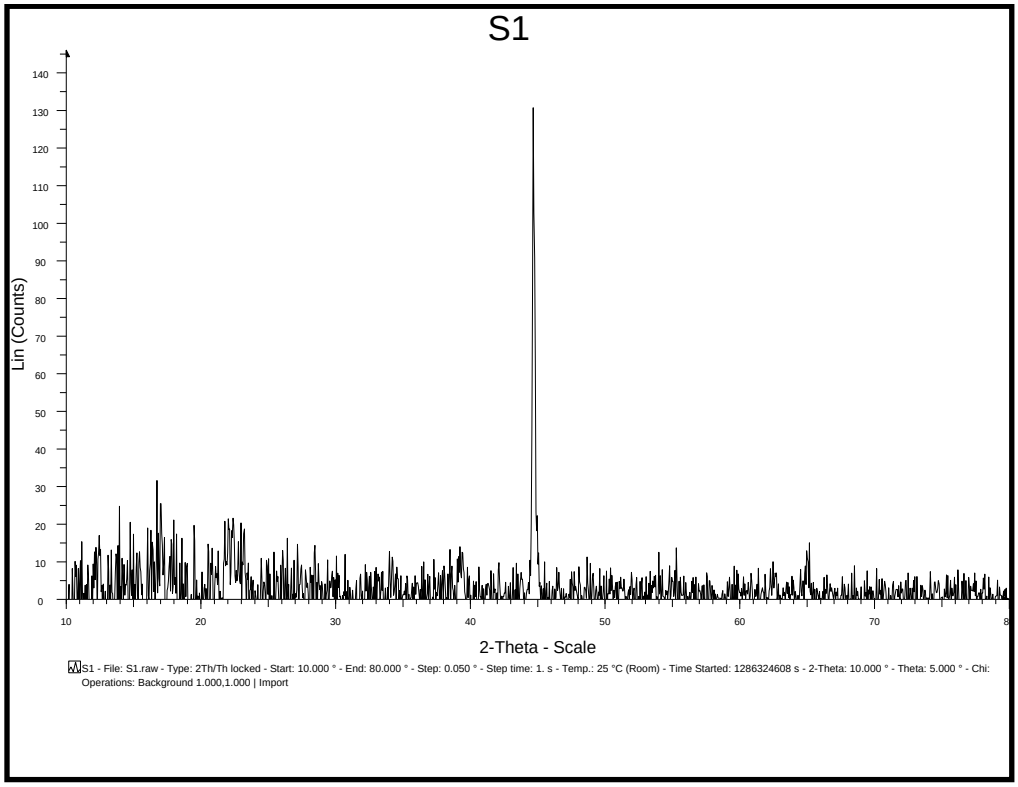


Figure 43: EVA Phase Identification Result of Sample 1

Result of X-ray Diffraction for Sample 2:

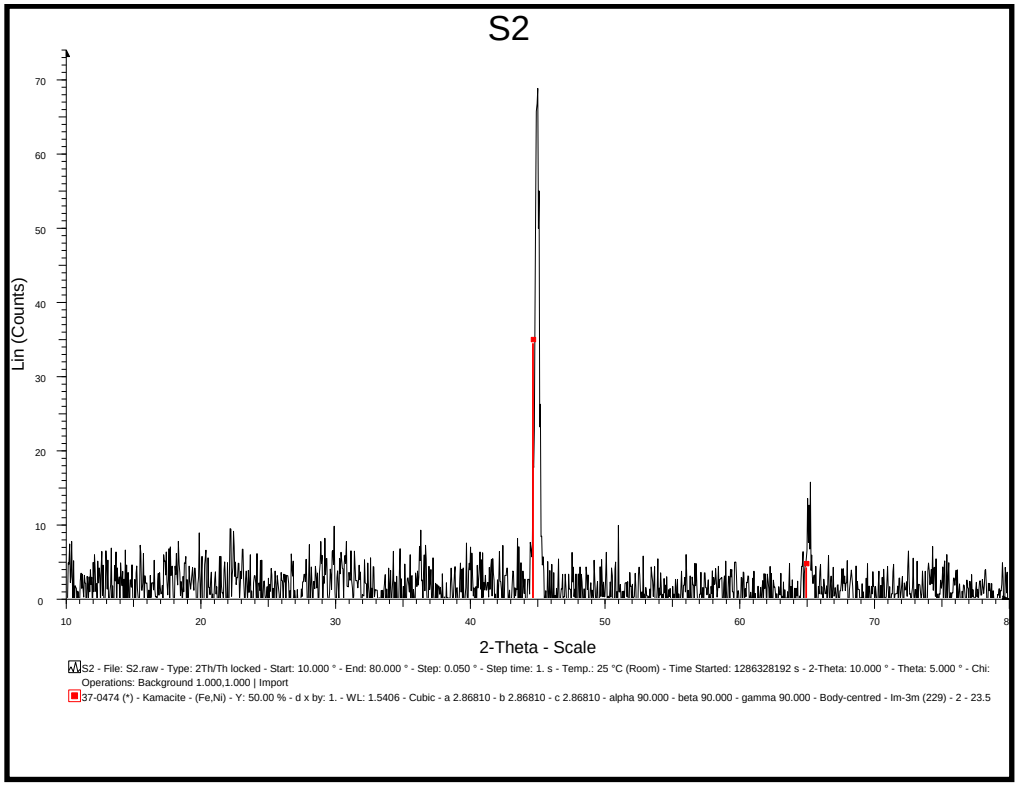


Figure 44: EVA Phase Identification Result of Sample 2

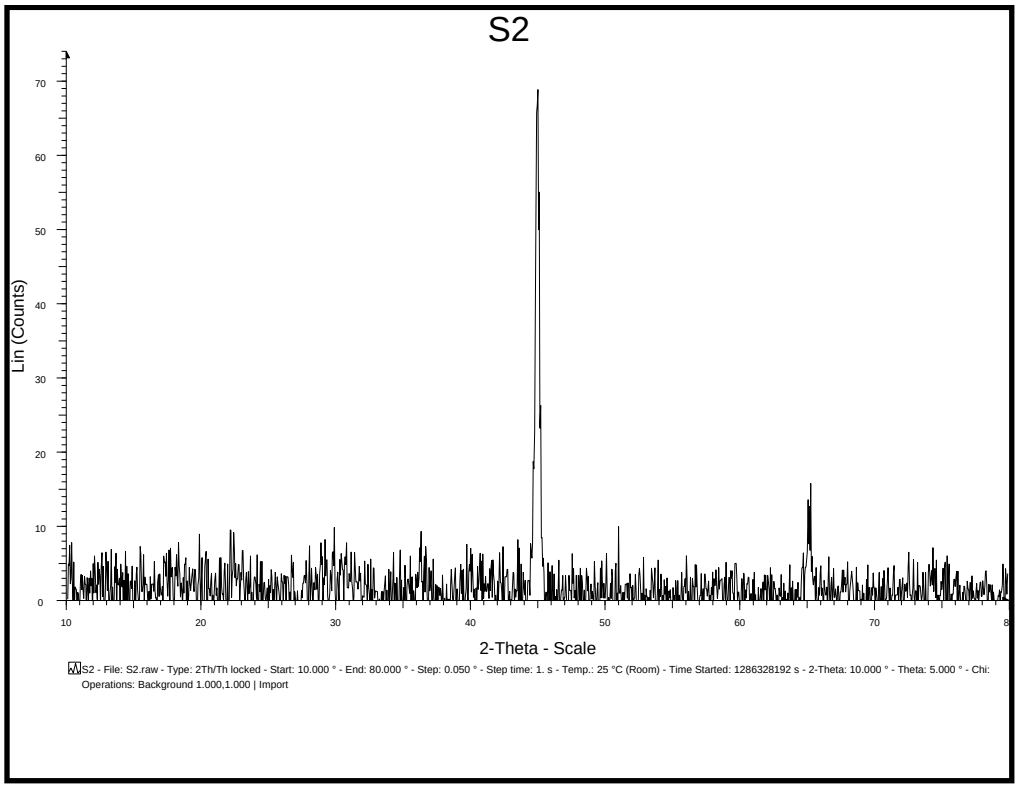


Figure 45: EVA Phase Identification Result of Sample 2

X-ray Diffraction result is in qualitative compare to hardness result which is quantitative. For this test, the wavelength that been used is 15406 wavelength. The limitation of X-ray Diffraction is the light element which sometime it cannot show the result of other elements. X-ray Diffraction will help to determine whether the elements are exist in unknown sample which in this case; the keris blade from Kuala Kangsar and Perlis. From the Phase Identification Result for both Sample 1 and Sample 2, the result show that the existence of Kamacite element (Ferum, Nickel) in the keris blade. The crystal structure is in cubic type and the type of structure is body-centered.

4.5 Recommendation

For future research, in order to get better result on microscopic examination, SEM should be added. SEM will help on the study of electron image of the keris blade and also give the information on the sample topography. Besides that, to confirm the phase identification results, and to obtain semi-quantitative phase amount estimates consistent with the actual elemental composition of the sample, combined X-ray Diffraction (XRD)-X-ray Fluorescence (XRF) analysis can been performed with EVA. EVA enables semi-quantitative analysis based on the reference-intensity-ratio (RIR) method using both XRD as well as XRF data simultaneously. The XRD results only help us to determine whether the elements are exist in the keris blade. Meanwhile, XRF results will help us to get the percentage of every element that exists in the keris blade. Some recommendations that can be made to keris makers are to use a power hammer for a good and uniform force during forging. For example, according to American Blade smith Standard, to get uniform force in process of shaping the keris, the keris makers should use different hammer. 1lb hammer used to straighten the blade, 8lb hammer used to forge the tang and 3lb hammer to refine. To get a pattern on the Damascus steel the keris makers can use the pressing process. For material, it is suggested by the American Blade smith Standard to use the 1080 or 1084 and 15N20 because these two alloys are very similar. 1080 is a course plain carbon steel and the makeup of 15N20 is nearly identical. It will have similar expansion and contraction coefficients. It also expands relatively the same when heated. Use borax as flux during metallurgy. A mixture of borax and ammonium chloride can be used as the flux.

CHAPTER 5

CONCLUSION

Further research will be done in order to get a better result of microstructure of the keris blade. Other Non-Destructive test also should be included so that more data gathered to get a better result of the metallic characteristics of the keris blade. Besides that, the study on the mechanical properties of the keris blade especially the hardness is vital as hardness is the most important aspect of a good keris. Optical Microscopy is used to study the metallographic, microstructure as well as optical texture of the keris blade. Meanwhile, X-ray Diffraction is to study on the crystallographic phases of the keris blade. It will determine whether the elements which are Ferum and Nickel are existing in the keris blade. Microhardness test will be run through this project to get the data on the hardness of the keris blade.

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