SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

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PHYSICAL AND CHEMICAL CHARACTERIZATION OF TIN IN AMANG FROM WEST COAST PENINSULAR MALAYSIA

By

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DECLARATION

I hereby declare that I have conducted, compiled the research work and written the dissertation entitled **"Physical and Chemical Characterization of Tin in Amang from West Coast Peninsular Malaysia"**. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any .other examining body or University.

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PENCIRIAN FIZIKAL DAN KIMIA TIMAH DALAM AMANG DARIPADA PANTAI BARAT SEMENANJUNG MALAYSIA

ABSTRAK

Tujuan kajian ini adalah untuk mencirikan dan mengenal pasti timah dari amang dari pantai barat Semenanjung Malaysia. Sampel untuk kajian ini diperolehi daripada dua tempat yang berlainan, iaitu dari Gunung Paku dan Lembah Kinta, Perak. Terdapat tujuh kaedah pencirian telah dilakukan dalam kajian ini seperti analisis taburan saiz zarah, kandungan lembapan, kehilangan dalam pembakaran, analisis komposisi unsur (menggunakan XRF), pengenalan fasa (menggunakan XRD), kajian mikroskopi (menggunakan SEM-EDX dan mikroskop optik) . Dari kajian ini, kedua-dua sampel menunjukkan kewujudan cassiterite (SnO2) dan mineral berat lain seperti ilmenit (FeTiO3), rutil (TiO2), hematit (Fe2O3) dan monazite [(Ce, La, Y, Th) PO4]. Selain itu, sampel dari Gunung Paku menunjukkan kandungan sulfur dan arsenik yang tinggi. Dirujuk kepada analisis liberasi, bagi saiz -75 µm, peratusan partikel yang terbebas untuk sampel dari Lembah Kinta adalah 85.91% manakala untuk sampel dari Gunung Paku adalah 80.24%. Oleh Itu, sampel daripada Lembah Kinta adalah lebih terluluhawa berbanding daripada sampel dari Gunung Paku. Terdapat beberapa mineral yang terlekat antara satu sama lain terutamanya dalam saiz zarah yang kasar. Kebiasaannya, sifat kristal yang didapati daripada kedua-dua sampel adalah dalam bentuk yang tidak teratur seperti ilmenit dan monazit. Berdasarkan analisis assay timah, peratusan kandungan timah di dalam sampel dari Gunung Paku adalah 0.44% dan tidak menguntungkan untuk diproses manakala untuk sampel dari Lembah Kinta, peratusan kasiterit yang ditunjukkan adalah 0.97% dan ekonomik untuk diambil.

PHYSICAL AND CHEMICAL CHARACTERIZATION OF TIN IN AMANG FROM WEST COAST PENINSULAR MALAYSIA

ABSTRACT

The purpose of this research is to characterize and identify the tin from amang from west coast peninsular Malaysia. There are two different places of samples obtained in this research, which from Gunung Paku and Lembah Kinta , Perak. Seven methods of characterization were implemented in this research such as particle size distribution, moisture content, loss on ignition, elemental composition (using XRF), phase identification (using XRD), microscopy study (using scanning electron microscopy, energy dispersive X-ray and ore microscopy) and tin assay. From this research, both of samples shown the existence of cassiterite (SnO_2) and other heavy minerals such as ilmenite ($FeTiO_3$), rutile (TiO_2), hematite (Fe_2O_3) and monazite [(Ce, La, Y, Th)PO_4]. Besides, sample from Gunung Paku shown high contamination of sulphur and arsenic. Referred to the liberation analysis, for -75µm size fraction, the percentages of liberated particles for the sample from Lembah Kinta was 85.91% while for sample from Gunung Paku was 80.24%. Therefore, the sample from Lembah Kinta was slightly more weathered than sample from Gunung Paku. There were some interlocking mineral with each other especially in coarse size particles. Most of the crystal habits were found for both samples are in irregular shape such as ilmenite and monazite. Based on the tin assay analysis, the percentage of tin content inside the sample from Gunung Paku was 0.44% and not profitable to be recover because less than 0.5% while for sample from Lembah Kinta, the percentage of cassiterite was 0.97% and it was economical to be extract.

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND PROJECT

Malaysia is a federation of thirteen states, eleven in the Malay peninsula (forming peninsular or west Malaysia), Sarawak and Sabah are separated by the South China Sea (Figure 1.1).

Figure 1.1: Location of Malaysia Within South-East Asia

Tin mining one of the of the firm that can be trace back to the Bronze age and have been done since the ancient times. Mining for tin takes place exclusively in West Malaysia, which straddles a zone of tin mineralization (the South-East Asian Tin Belt) streching from Indonesian islands of Bangka and Belitung to Thailand (Tompkins, 2003).

The Malay peninsula is divided into two geological povinces by a central of granite mountains. To the east of this range, tin ore is usually in hardrock form (primary sources), while to the west, the tin ore is tends to be alluvial form (secondary sources). Tin is recovered from these as cassiterite (SnO_2), from which it is extracted by smelting. The most well-known technique in recovery of cassiterite is hydraulic-gravity processing by using jigs or sluices boxes as in Thailand, Bolovia and Malaysia. This processing technique is believed to be dregs effective with increasing in fineness and decreasing in density of the heavy minerals (Funtua, et. al, 1997). However, this method is comparatively cheaper than the chemical method such as leaching and froth flotation.

Historically, the oldest industries in Malaysia is tin mining and it was a dominant contributor to the country economy. There was rapid advancement of tin industries in Malaysia in the mean of 19th century, making Malaysia become one of the largest tin producer of the world. However, after the collapse of the world tin industry in 1985, tin industries in Malaysia are no longer productive. Although no longer active, the reservoir of tin tailing in these fields generates momentous interests. These tin tailing known as 'Amang' are process to reclaim the tin from tailing.

1.2 SIGNIFICANT ON PROJECT

This research mainly focus on the characterization of tin ore from amang though seven different methods. Amang, left abundant in the tin fields are rich in heavy minerals containing rare earth elements and also contain tin that escape to the tailing during tin recovery process. This give a significant economic interest in amang for recovery of tin and heavy minerals. The significant of these characterization studies is to determined on the samples will furnish a fundamental vital data on the existances of tin in the amang and to investigate either that tin exist in the sample are economical or not to be recover.

1.3 PROBLEM STATEMENT

Amang is by product from tin mining and contained mixed of heavy minerals Usually the heavy minerals associated in amang are ilmenite, zircon, rutile, monazite, and xenotime. Amang also contained significant percentages of cassiterite which is the main source of tin. Previously, there are no further research for characterization of tin in amang, most of the research only characterize the rare earth mineral. Visual observation on the sample is impossible to identify the present of cassiterite inside the sample and also difficult to distinguish all or related constituents of mineral. It is also inconceivable to clearly decide the amount of mineral present in the amang without advance characterization. Therefore, the further characterization of samples obtained were be done to permit the strong evidence of the type of mineral present especially the present of cassiterite. Futher characterization also been done to determine either that cassiterite exist are economical or not to be recover for the sample from G unung Paku and Lembah Kinta.

1.4 OBJECTIVES

The objectives of this research are as follows:

- To characterize amang samples from Gunung Paku and Lembah Kinta, Perak according to different size fraction
- To identify the associate mineral and what is available within amang samples

1.5 SCOPE OF RESEARCH

Bulk characterization and different size fraction characterization of tin particle ore are the main characterization for this study scope. These bulk characterization analysis included sieve size analysis, elemental compostion analysis (XRF), phase identification (XRD) and morphological study using optical microscope and SEM-EDX.

Amang samples from diffferent places were undergoes these characterization after doing sieve size analysis with the obtained size of (+5.00)mm, (-5.00,+4.00)mm, (-4.00,+2.80)mm, (-2.80,+2.00)mm, (-2.00,+1.40)mm, (-1.40,+1.00)mm , (-1.00,+0.600) mm, (-0.600,+0.500) mm, (-0.500,+0.355) mm, (-0.355,+0.250) mm, (-0.250,+0.180) mm, (-0.180,+0.125) mm, (-0.125,+0.075) mm (-0.075) mm for sample from Gunung Paku and size of +0.425mm, (-0.425 +0.300)mm, (-0.300 +0.200)mm, (-0.200 +0.150)mm, (-0.150 +0.106)mm, (-0.106 +0.075)mm, (-0.075 +0.053)mm, -0.053mm for sample from Lembah Kinta.

The selected size of these samples were study for their properties that may exhibit by optical identification and mineral liberation observation under polarizing ore microscopy and scanning electronic microscopy (SEM). With the help of EDX, the semi-quantitative weight percentage of selected particle were detected. The SEM images also were utilized in the analysis of liberation percentage at each fraction.

1.6 DISSERTATION ORGANIZATION

Five chapters which are introduction, literature review, methodology, results and discussion and last chapter of conclusion and recommendation are the main content in this thesis. Chapter one which is introduction, are telling about the research background and the aim to do this work. Next chapter, is literature review on previous works and information about the works regarding the work project.

Chapter three shows how to do these project in a sequences way starting from the early project until get the answer and fulfill the objective research. Most important chapter is results and discussions are listed in chapter four. All works that done on previous chapter are come out in chapter four and reasons for these results. Finally, the conclusion is made regarding the results performance in this research and the recommendation for the future work being explained in the last chapter which is chapter five.

CHAPTER 2

LITERATURE REVIEW

2.1 GEOLOGY

The sample utilized in this research were obtained within west coast of peninsular Malaysia. The first sample is from Gunung Paku and the second sample was collected from Lembah Kinta.

2.1.1 General Geology And Classification of Gunung Paku

Gunung Paku is mainly associated with widespread occurance of sheet-like quartz veining systems parallel to the strike of the host rocks and confined with narrow N-S trending fault zone (Ariffin, 2009). Figure 2.1 show the general geology of the Gunung Paku. The mineralization formed within a thick sequence of metasedimentary rock that belongs to the Baling Palaeozoic Age.

The host rock pf weakly metamorphosed argilite experienced strong tropical weathering that resulted in a thick sequence of light brown to light grey oxidized profile. The mineralized veins range from simple quartz-cassiterite, quartz-tourmaline-cassiterite to complex quartz-cassiterite-polymetallic sulfide veins. Wall rock alterations at Gunung Paku are mainly consisting of silicification, tourmalinization chloritization, sericitization and kaolinization; normally adjacent to mineralized quartz veins and brecciated-fault gouge zones (Ariffin, 2009).

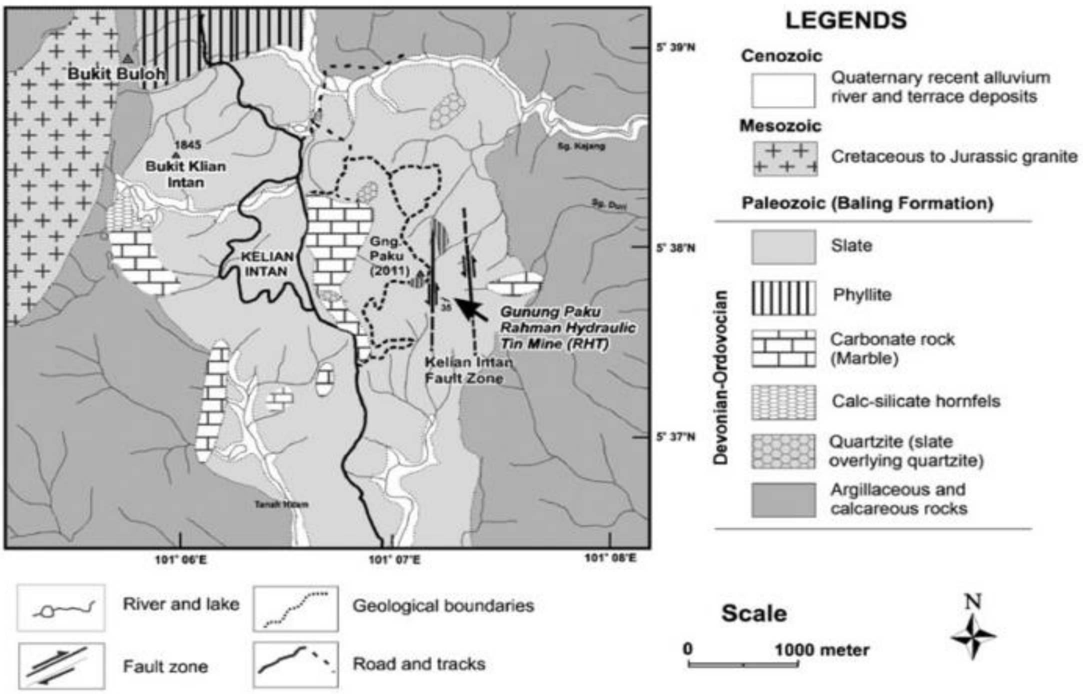

Figure 2.1 : Geological map of the Gunung Paku tin deposit (Ariffin, 2009)

The veins are categorized as barren quartz veins, tourmaline cassiterite quartz veins and complex sulfide tin-rich quartz veins. Rutile, pyrite, chalcopyrite, arsenopyrite, cassiterite, scorodite, trippkeite, and covellite are usual metallic minerals that went with the tin mineralization. The primary tin sources had been subjected to deep and intense tropical weathering to form thick alluvial deposit (Ariffin, 2009).

2.1.2 General Geology and Classification of Lembah Kinta

Limestones constituted the bedrock over the greater portion of the Lembah Kinta. It formed striking hills rising from the alluvial plain which is the most common rock in the calcareous series. Limestone deposited in a clear sea but during the period of deposition, muddy and sandy allows the limestone to crytalline into marble, and the argillaceous beds to schist and quartzite respectively.

Arillaceous and arenaceous were formerly though to be part of the Triassicsystem. The southwestern part of Tronoh there are sedimentary beds which form part of the quartize range exposed on the Seputeh and Parit Road west of Seputeh beyond the area mapped.

Granite which is forms the Main range and the Kledang Range has been intruded into sedimentary rocks of known Carboniferous and Triassic age in other parts of Malaysia. The granite is thus post-Triassic and form evidence obtained by Dutch geology in Borneo it is probable that it was intruded in late Mesozoic time, possibly during the Createaceous period.

The whole of Lembah Kinta floor is covered by alluvium and is also widespread along the river valleys in the granite hill. It is not depicted on the map, for the nature of the bedrock beneath, it is generally known either from exposures or from samples obtained by boring carried during prospecting work.

On the eastern side of the valley near Gopeng not only the plain covered alluvium, there are alluvium of the valley. These hillrocks approximately reach 80 meters above sea level and the beds forming them were probably deposited most of the alluvium of the valley had been laid down (Ingham, 1960).

2.2 AMANG

Amang is a term that are widely accepted in Malaysia for the mixed of heavy minerals which remain as by product after tin oxide (cassiterite) has been extracted from tin ore. During the processing of tin ore, heavy minerals that are channeled to the tailing contain some tin mineral, cassiterite ($Sn0_2$). After some period of time passed, these heavy minerals and tin accumulated in the amang. The reservoirs of amang generate significant economic interest. This due to the present of valuable minerals in amang that are economically beneficial to the mineral industries.

Other than tin, amang also comprise of various heavy minerals which are valuable to be recover. Heavy minerals refer to the minerals with specific gravity (SG) greater than the SG of quartz which is 2.7 g/cm^3 (Reyneke & Van Der Westhuizen, 2001). Heavy minerals that are commonly exist in amang are rutile (TiO_2), xenotime (YPO_4), monazite ([Ce,La,Nd,Gd,Th] PO_4), ilmenite ($FeTiO_3$), Zirconia ($ZrSiO_4$) and struverite ($Nb.Ta.TiO_2$).

2.3 TIN MINERAL

Tin is one of the few metals that have been used since the Bronze Age. In passed of times, tin was used to alloy with copper to make bronze that haveimprove properties compare to pure copper. Tin is chemically inert metal that are ductile, soft, malleable silvery-white in color and it has highly crystalline structure. Specific gravity for tin is 7.31 g/cm^3 and it has a low melting point of 232 ^oC. It Mohs scale of hardness is between

6 to 7. Major usage of tin is in making tin plate and as a solder for electonic parts, machinery and plumbing. It is highly resistant to corrosion and used as a coating to cover of other metals.

In nature, tin does not ensue as native element. It occur in oxide mineral like cassiterite (SnO_2) or in various sulphides minerals such as teallite ($PbSnS_2$) and stannite ($CuFeSnS_4$). Generally, the main source for tin is from cassiterite. Cassiterite in the earth crust is sufficiently abundance making it the only tin mineral that have economic value. In it chemically pure state, cassiterite contains about 78.6 percent tin (Falcon, 1982). But this is rarely condition and usually tin content in cassiterite varies between 73 to 75 percent.

The source of cassiterite can be found in two types of tin deposits. The first type of deposits are known as hard rock deposits, the cassiterite associated with intrusions of granites and occurs as primary accessory constituent. The tin veins and ore bodies located at contact zones of tin-bearing granites and others rocks (Ariffin, 2009). Tin deposits can be classified into three types of tin-bearing mineral assemblages which are stanniferous pegmatites, quartz-cassiterite and sulphide-cassiterite (Taylor, 1979)

Stanniferous pegmatites formed in the areas where mineralization is associated with deep-seated intrusions of acid granites. The pegmatite consist of the quartz-microline type. The cassiterite mineral is irregularly spread through the pegmatite body as large black or dark brown dipyramidal crystal. This type of deposit is mined in the Republic of Congo, and Nigeria.

The quartz-cassiterite deposits in the same areas as stanniferous pegmatites deposits and occurs with the same acidic granitiod intrution. The cassiterite commonly associated with wolfromite and consist of regular quartz veins, and stockworks. Most of the tin deposits in the southeastern Asian consist of the quartz-cassiterite type.

The sulphide-cassiterite deposits are rich in sulphides of silicates that rich in iron. In occurance depend on the first two type of deposit. This type of deposits includes the skarn deposits of cassiterite associated with chlorite, arsenopyrite, and pyrholite, tourmaline, hydrothermal tin-silver deposit and hydrothermal tin-lead-zinc deposits.

The second types of tin deposit is placer deposits, also known as secondary deposit. Placer deposits become the most source of tin produtction from South East Asia. There are two types of places deposits which is alluvial and elluvial deposits. Both types of deposits are econimcally vital and provide up to 70 percent of the world tin supply. The formation of placer deposits is from erosion of the primary tin deposits. The alluvial tin deposit is one of the main source for tin mining in Malaysia.

2.4 HEAVY MINERAL ASSOCIATED WITH AMANG

As mention earlier, valuable minerals in amang was categorized as heavy mineral and have a high specific gravity which is higher than specific gravity $2.7 g/cm^3$ of quartz (Reyneke & Van Der Westhuizen, 2001). The specific gravity of valuable minerals in amang usually higher than 4. The heavy minerals occur in various igneous and metamorphic rocks in low concentrations.

Heavy minerals are mechanically resistant to weathering and chemically stable. Due to this properties, the heavy minerals will accumulate in inner bank of meandering river channel and along coastal of shorelines. This is because flows of water is faster in outer bank of a meander. The light mineral will travel more compare with heavy minerals. Then, deposition of heavy minerals in inner bank of meander will formed.

2.4.1 Ilmenite

Figure 2.2 : The picture of Ilmenite from Miass, Ural Region, Russia

Ilmenite ($FeTiO_3$) is a titanium-iron oxide mineral with idealized formula. It color is weakly magnetic black or steel-gray. It is the most momentous ore source for titanium. Specific gravity for ilmenite was in range 4.5 to 5.5 and it has a Mohs scale hardness of 5 to 6. Ilmenite is concentrated into layers by a process called “magmatic segregation” and formed as a primary mineral in mafic igneous rocks. Magmatic segregation is a general term referring to any process by which one or more minerals become locally concentrated (segregated) during the cooling and crystallization of a magma.

The heavy crystal of ilmenite are deposited to the bottom of the magma chamber and form layers. As a result, an ore body formed with titanium rich and it can be found in Russia, Brazil, Canada, Sri Lanka, Norway, Australia, China, South Africa, Malaysia, Thailand, India, Sierra Leone, and the United States.ilmenite is mined from sand that formed from weathering of ilmenite-bearing rocks. Ilmenite also present in amang and it constitutes the major minerals in amang up to 80 percent (Yasir et al, 2007)

As titanium usage is increasing, ilmenite become important to the industries. Ilmenite sand are used to clean diecasting dies in sandblasting. Ilmenite ore are commonly used as a flux to line the blast furnace hearth refractory in steel making. Titanium dioxide is used in paint industry as a base pigment due to the good endurance, high opacity and great luster. Beside, titanium oxide are used to provide colour as pigment in ceramic, paper, plastics, and cosmestics. The application of titanium alloys are used in aircraft parts, surgical implants and in high-performance alloys due to it properties. Titanium has a properties of non-corrosive, light weight, high melting point (>800 ^{o} C) , and has a good strength.

2.4.2 Rutile

Figure 2.3:The picture of wine-red rutile crystal from Stony Point, North Carolina

Rutile is an oxide mineral made principally out of titanium oxide with idealized formula (TiO_2). It is a typical embellishment mineral in igneous rock, essentially in granite and pegmatites, and in metamorphic rocks. It is the most stable polymorph of TiO_2 at all temperature and is a wine-red crystalline. Rutile or Titanium Oxide, (TiO_2) mineral is used in creating ceramics in high refraction options, and of course refined to produce titanium.

It has specific gravity of 4.2 to 4.4 and a Mohs scale hardness of 6 to 6.5. It form an vital constituent of heavy minerals deposits and one of the minerals recover from heavy minerals deposit for it value. Rutile varies in color includes metallic-gray, golden-yellow, brownish-red, dark-red and reddish-black.

Rutile is one of the favored minerals for the generation of titanium dioxide white pigment, predominantly through the chloride producing process. Titanium minerals are monetarily important because it is a source in titanium metals production through Kroll process. Titanium has light weight, good strength, and good thermal and corrosion resistance. In industries, the usages of Rutile are similar to the Ilmenite because both minerals contain titanium.

2.4.3 Monazite

Monazite is a primary ore of several rare earth metals such as cerium (Ce), lanthanum (La), and thorium (Th). All these metals are considered quite beneficial and have distinct industrial uses. Thorium is an exceedingly radioactive metal and could be utilized as a substitution for uranium in atomic power generation. Also, it can be utilized as a part of recalcitrant application, light mantles, aviation composites and welding electrodes.

Cerium can be utilized as an catalytic converter for the oxidation of carbon monoxide (CO) outflows in the exhaust gases from vehicles. Cerium mixes are additionally utilized as a part of the fabricate of glass, both as a segment and as a decolourizer (cerium 2014). Monazite occurs elsewhere in peninsular Malaysia. It acquired from weathering of granitic rocks and present in a small amount throughout the alluvial deposits.

The colour of monazite commonly orange-brown or yellow-brown glassy properties. It is opaque but small crystall can be transparent. The specific gravity of monozite is between 4.6 to 5.7 and it Mohs hardness scale in range 5 to 5.5. Monazite is non conductive and non magnetic minerals. Monazite is radioactive, and speciments are generally metamict, a mineral has become virtually amorphous owing to the breakdown of the original crystal structure by internal bombardment with alpha particles (helium nuclei) emitted by radioactive atoms within the mineral.

Figure 2.4: Monazite-(Ce) from Sao Joao da Chapada, Diamantina, Minas Gerais

2.4.4 Zircon

Zircon is the major source of zirconium and it is a nesosilicates mineral. Zircon has colossal application in different industries, for example, foundry, earthenware production, unmanageable and as gemstone. Zircon has high hard-headed with a liquefying point of 2430^{o}C. it is utilized as a part of liquid metal form, foundry sand shape and steel throwing spouts. In artistic industry, it been utilized as earthenware coats.

Zircon has specific gravity of between 4.6 to 4.8 and a Mohs scale hardness of 7.5. Zircon differs in colour with the most widely recognized colour is dark brown. The colour of zircon incorporates gray, brownish-red, pink, black, light blue, and colorless. Zircon happens fundamentally in volcanic rocks, more often than not in pegmatites. It additionally happen in metamorphic rocks and sedimentary rock as dentrital grains. It frames huge financial fixations inside the substantial mineral deposits.

Figure 2.5 : Zircon Natural Chips from Brazil

2.4.5 Xenotime

Xenotime is a main source of yttrium and the major use of yttrium is in the phosphor powders for low energy lighting. Yttrium is utilized to make yttrium-aluminum garnet lasers and yttrium iron garnet microwave channels. It is likewise used to make cubic zirconia gems, in fighter jet engines, medical, realistic innovations, in electronic segments for missile defense systems and others. Some of xenotime might be radioactive because of uranium and thorium contaminations.

The colour of xenotime incorporates greenish dark colored, yellow, quieted red and gray. It is vitreous to resinous. The specific gravity around 4.4 to 5.1 and it Mohs hardness is around 4 to 5. The streak is in pale darker to yellow in color. The associated minerals of xenotime as a rule are quartz, feldspars, and mica (biotite)

Figure 2.6: The picture of xenotime from Novo Horizonte, Brazil

2.5 CHARACTERIZATION OF AMANG

The characterization of amang is fundamentally vital data required for recovery of heavy minerals from amang have been done. Reyneke and Van Der Westhuizen (2001) had described the standard technique utilized for the characterization and preparation mineral-bearing sand samples. This standard method can be applied for characterization of the amang samples. The method included particles size distribution, element composition determination, phase identification, and microscopic study.

2.5.1 Particle Size Distribution (PSD)

Particle size distribution (PSD) is critical information with a specific end goal to decide the liberation size of the minerals and it shows the ranges of sizes in the minerals samples. The PSD gradient will decide the distribution pattern in an sample either the size of particles in sample are single size or varied with size range. Steep slope show little size range of mineral sample or called single size. At that point, less steep slope in PSD graph data shows minerals differed with size range.

Additionally, the estimation of percentiles in graph will indicate the statistic of size dissemination of an samples. It gives the data about how the size dissemination are spread over interim from littlest value to the biggest value. The $D_{25,}$ $D_{50,}$ $D_{75,}$ values represent the 25^{th}, 50^{th}, and 75^{th} percentile of cumulative passing size respectively. 25^{th} percentile is known as first quartile, 50^{th} percentile as median and 75^{th} percentile as third quartile.

Uniform coefficient, C_u can be calculated in order to determine the gradation of the sample. the C_u value is in excess of 6 implies the the sample is well graded. Sample contain particles of extensive variety of sizes and has a decent portrayal of all sizes. On the off chance that C_u is under 6, it implies that does not have a good portrayal of all sizes of particles. Sample generally has single size particles. ("Soil gradation," 2014)

$$C_u = \frac{D60}{D10} \tag{2.1}$$

Where

D10 = passing size of particles at 10%

D60 = passing size of particles at 60%

2.5.2 Phase Identification Using X-Ray Diffraction (XRD)

X-ray diffraction (XRD) is utilized to recognize the phase of a chemical compound from its crystalline structure. X-ray beams are electromagnetic waves with high recurrence and short-wavelength. In a X-ray tube, filament is heated to deliver electrons. The high voltage in X-ray tube enables the electrons to accelerate toward the target in vacuum. Consequently, rapid electrons are besieged to an target with high energy. X-ray beams are created after the impact of fast electrons to the target. Less than one percent of the energy is changed into X-beam and others are changed over into heat. In this way, broad cooling is required to the X-ray tube.

Energy is transfer into the target when the rapid electrons are bombarded to the target. At the point when electron of a atom's inward has adequate energy, it will energized and transition of electron will happen. Electron hops from lower to higher energy level. As the electron falls, energy will be discharged by radiating photon with particular energy. In other words, X-ray with particular wavelength is created. The wavelength is investigated and deciphered by the framework.

$$n\lambda = 2d \sin\theta \tag{2.2}$$

Where

λ = The wavelength of the X-ray

d = The spacing of the crystal layers

θ = The incident angle

n = An integer

Based on the Bragg's law equation (2.2). The information on the spacing between atomic planes of a crystal can be obtained when construction interference is detected at a given incident angle and a wavelength of the incident beam. The incident ray must be travel in-phase. When the spacing between atomic planes is obtained, crystal structure of a materials can be determined (Leng, 2010).

2.5.3 Elemental Composition Using X-Ray Fluorescence (XRF)

The purpose of XRF analysis is to recognize the mineral composition present in sample. There are wavelength dispersive spectroscopy (WDS) and energy dispersive spectroscopy (EDS). The fundamental distinction between WDS and EDS are on the instrumentation in the machine. Both of the technique can be utilized for the sample analysis. High energy X-beam will bombarded from the source and excites the atoms in a sample. Photon will be produced from the surface of sample and recognized by detector. Data collection and processing system will distinguish the present of mineral in an sample

Loss of ignition (LOI) is utilized to determine the level of organic substance in the samples. Temperature around $600^{O}C$ is applied to permit dissipation of volatile materials and organic substance due of low breaking point of those two materials. After the start, every one of the minerals stay in the example. The distinction in weight will indicates the measure of natural substances and unstable materials. The percentage of LOI is used in XRF elemental analysis.

2.5.4 Microscopic Study

2.5.4 (a) Ore Microscopy

The ore microscopy is the one of the basis equipment used for the petrographic observation of the substantial and monetaarily imperative group of minerals alluded to collectively as ore minerals. It is comparative with the ordinary petrographic microscope in the frameworks of focal point, analyzer, polarizer, and various diaphragms employed, but it contrasts in term of essential method of illumination. There is a l0ight source over the sample permit observation by reflected from polished surface (james, 1994).

The observation of ore microscopy are generally made either with Crossed Polarized Light (XPL) or with Plain Polarized light (PPL). The application of XPL allows the examination of anistropism and internal reflection while during the observation with PPL, minerals can be determined by its hardness, color, reflectance bireflectance and reflection pleochroism. Isotropic minerals are minerals that have the equal properties in all directions. This means light passes through it in the same way, with the same speed, no matter what direction of light is travelling. Anistropy minerals have contrast properties in distinct directions of light. Therefore, the light travels through it in different ways and different velocities, rely upon the direction of travel through a grain. Isotropic and anisotropic minerals are most of the time, easily distinguished because isotropic minerals do not transmit ligt (always black) when viewed under Crossed Polarized Light (XPL).

2.5.4 (b) Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) with energy dispersive X-ray (EDX) is utilized to analyze the liberation size of particular mineral. SEM employs electron beam directed to the specimen surface. The structures of SEM consist of electron gun, condenser lens and vacuum system. Electron gun produces electrons by heating the tungsten filament and accelerates electrons to energy about 2kV to 40kV

After the incident beam strike the surface of specimen, SEM normally can detect secondary electrons and backscattered electrons. Both methods are commonly used as a signal in SEM. It detected by a scintillator-photomultiphier known as the Everhart-Thornley detector. The photomultiplier converts the photons into pulses of electrons and then amplified the intensity to the cathode ray tube (CRT). A clear picture of specimen can be seen on the monitor screen.

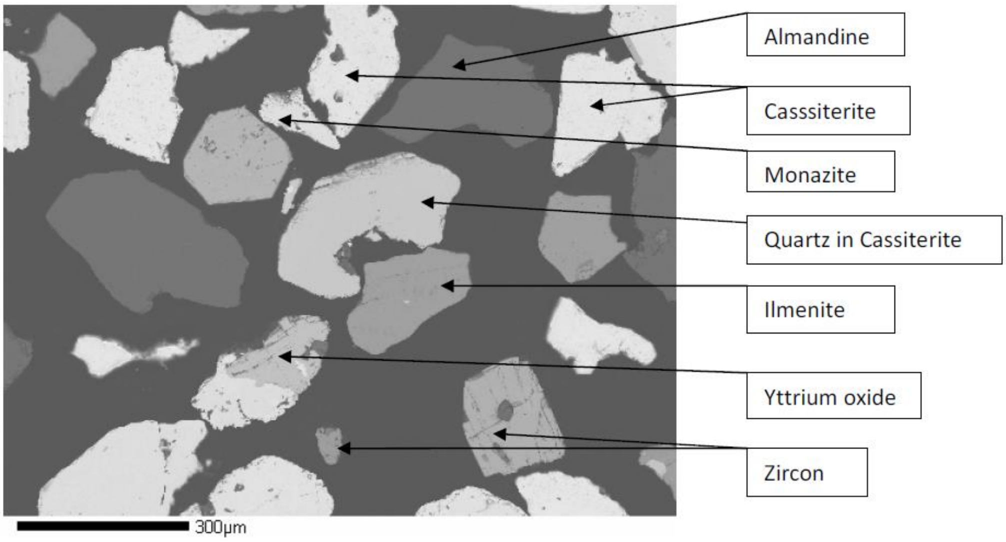

Figure 2.7: Example of SEM image of representative cassiterite sample (Ogwuegbu et al., 2011)

At the same time, X-ray will also produce from specimen when the incident beam strikes the surface of specimen. X-ray emitted from the specimen shows the characteristic of atoms present in the specimen. This is because every element has specific wavelength and allow the machine to match with reference wavelength. Therefore, a qualitative analysis can be carried out (Goodhew, 1988).

Backscattered electrons (BSE) comprise of high-vitality electrons beginning in the electron beam, that are reflected or back-scattered out of the specimen interaction volume by elastic scattering interactions with specimen atoms. Since substantial elements (high nuclear number) backscatter electrons more firmly than light elements (low nuclear number), and along these lines seem brighter in the picture, BSE are utilized to distinguish balance between regions with various chemical compositions (Goldstein, 1981).

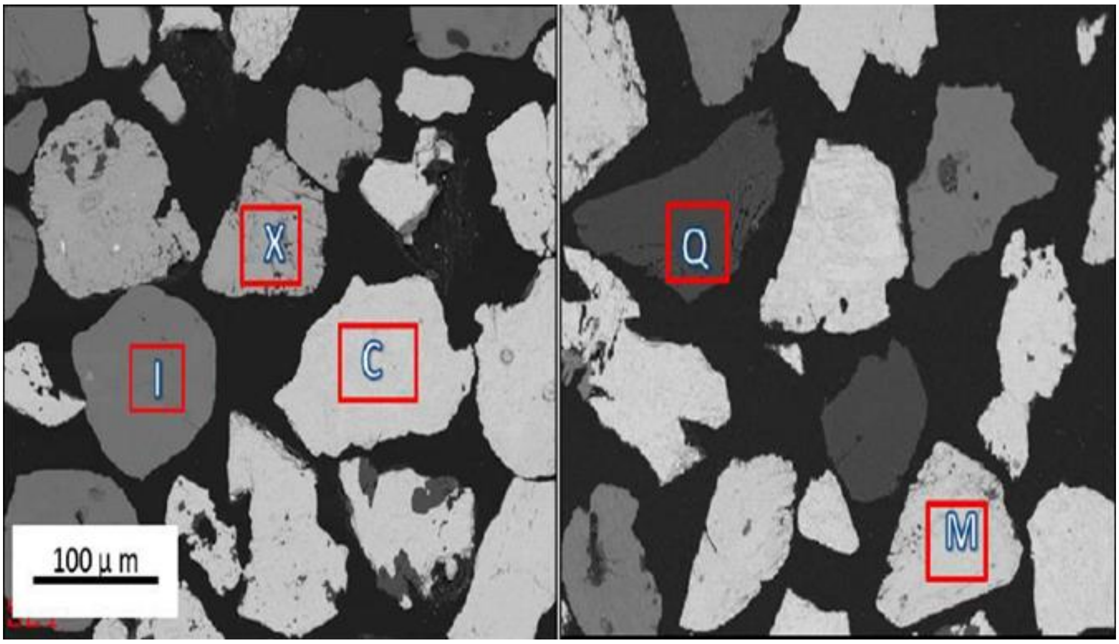

Figure 2.8:Figure 2.9: Example Back scattered SEM images illustrating M= Monazite, C= Cassiterite, X= Xenotime, I= ilmenite, and Q= Quartz (Harjanto, Virdhian, & Afrilinda, 2013)

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

In this chapter, a full description on sets of methods that govern the full scope of this research is outlined in order to achieve the objectives. This research on characterization is concerned on the degree of liberation of the amang samples from Gunung Paku and Lembah Kinta. Both physical and chemical characterizations of the both samples are further discussed.

3.2 MATERIAL

The sample of this research was obtained from Gunung Paku and Lembah Kinta and was used as a raw material.

Table 3.1 : Received sample from Gunung Paku and Lembah Kinta

Gunung Paku	Lembah Kinta
	
Top size : 5.79 mm Total weight of sample received: 1.45 kg Colour : Grey	Top size : 0.48mm Total weight of sample received: 0.64 kg Colour : Black