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INFLUENCE OF NaOH IN THERMAL REDUCTION OF WOLFRAMITE IN ORE MINERAL

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Abstract

The annealing process of ore mineral which is important for designing scientific and economic schedule. The samples were collected from Pharchaung Mine in Tanintharyi Region. The 70% wolframite sample (Fe,Mn)WO_4 was prepared by magnetic separation method and ore mineral of (Fe,Mn)WO_4 were collected in this mine. In this research work, to emphasize the value of considering wolframite as thermal reactions and chemical reactions methods. The behavior of structural tile were investigated using thermal analysis techniques. The weight loss of 70% wolframite sample (Fe,Mn)WO_4 was observed from TG curve and the phase transition occurred from DTA curve. The crystal structure of 70% (Fe,Mn)WO_4 powder and ore mineral of (Fe,Mn)WO_4 were observed by XRD and also the phase changes of this powder were investigated at 450°C and 500°C in three hour. And also demonstrated its application to provide a better understanding of the occurrence and origin of wolframite minerals. Aqueous solution containing sodium hydroxide (NaOH) was used as the alkaline leaching media and it was the most beneficial agent stabilizing the solid phase in the aqueous environment.

Keywords: Annealing process, TGA curve, Wolframite, magnetic properties.

Introduction

Wolframite, is a principal ore of tungsten. It is an iron and manganese tungstate mineral. It has a hardness of 5 to 5.5 mhos, specific gravity of 7.1 to 7.5, is dark gray, reddish brown, brownish black, or iron black in color. Wolframite is commonly found in granite and pegmatite dikes, and is often associated with cassiterite; it also occurs in sulfide veins and placer deposits. It has been suggested that the name ferberite be limited to mixtures containing not more than 20 per cent of the hubnerite molecule and the name hubnerite to those containing not more than 20 per cent of the ferberite molecule. This would leave the name wolframite for mixtures containing more than 20 per cent of both $FeWO_4$ and $MnWO_4$. Because heat causes tungsten to expand at about the same rate as glass, the metal is widely used to make glass-to-metal seals. Tungsten ore is a rock from which the element tungsten can be economically extracted. The ore minerals of tungsten include wolframite, scheelite, and ferberite. Materials processing is one of the most important and active areas of research in heat transfer today. With growing international competition, it is has become crucial to improve the present processing techniques and the quality of the final product. Heat transfer is extremely important in a wide range of materials processing techniques such as crystal growing, casting, glass fiber drawing, chemical vapor deposition, spray coating, soldering, welding, polymer extrusion, injection molding, and composite materials fabrication. Tungsten is an economically important metal, being widely used in light-bulb filaments, electron and television tubes, abrasives and special alloys such as tool steels. Tungsten carbide is of great importance to the metal-working, mining and petroleum industries. Contamination from these sources is, therefore, possible in industrial and urban areas. Scintillators for dark matter search, semiconducting photoelectrodes for photoelectrolysis or humidity sensors for meteorology are some of the direct applications that wolframite-type compounds with chemical formula AWO_4 present. When a material is heated its structural and

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chemical composition can undergo changes such as fusion, melting, crystallization, oxidation, decomposition, transition, expansion and sintering. Using Thermal Analysis such changes can be monitored in every atmosphere of interest. The obtained information is very useful in both quality control and problem solving. Thermal Analysis is the term applied to a group of methods and techniques in which chemical or physical properties of a substance, a mixture of substances or a reaction mixture are measured as function of temperature or time, while the substances are subjected to a controlled temperature programme. For specific application, the magnetic properties are important. Non-magnetic materials have to be used whenever the magnetic fields can be perturbed in radiation equipment or when shielding is positioned near electrical sensors.

Experimental Procedures

Preparation of pyrite samples

The 70% wolframite sample (Fe,Mn)WO_4 was prepared by magnetic separation method and ore mineral of (Fe,Mn)WO_4 were collected from Pharchaung Mine in Tanintharyi Region. Firstly ore mineral of (Fe,Mn)WO_4 was divided and collected from rock sample according to their raw group formations (colour). The 70% wolframite sample (Fe,Mn)WO_4 was grounded again and again to form homogeneous powder and also ore mineral of (Fe,Mn)WO_4 was grounded again and again to form homogeneous powder. The structure of these powder samples were determined by XRD holder to determine the structure and lattice parameter.

Most commonly, the mass change is related to sample purity or composition. The heat changes within a 70% wolframite sample (Fe,Mn)WO_4 was monitored by measuring the difference in temperature (T) between the sample and the inert reference. This differential temperature is then plotted against temperature or time to get DTA curve. TG and DTA was carried out using the DTG-60H thermal analyser at the Research Center , Yangon University.

The obtained The 70% wolframite sample (Fe,Mn)WO_4 was heated at 450°C and 500°C in furnace 3 hrs each and slightly cold down to room temperature. The samples were grounded again and again to form homogeneous powder. The structure of these powder samples were determined by XRD holder to determine the structure and lattice parameter. In chemical analysis, 6 g of natural ore (Fe,Mn)WO4 are grinding with mortar and pestel, then 2 g of sodium hydroxide solution are added. The mixture of this sample is grinding and then heated at 300 in one hour. And then, this mixture sample was annealed in 450°C, 500°C in furnace three hour each. The samples were grounded again and again to form homogeneous powder. The structure of these powder samples were determined by XRD holder to determine the structure and lattice parameter.

Results and Discussion

Structural characterization and TG/DTA analysis

More extensively, the ABX_4 compounds exhibit numerous crystalline forms and the phase transitions between some of them were shown to be of great interest .In this paper concentrate on the 70% (Fe,Mn)WO_4 compounds by magnetic separation method; respectively, with orthorhombic structure. In the literature, it is known that (Fe,Mn)WO_4 monoclinic structure crystallize in the wolframite structure when synthesized between 400°C and 500°C. As-obtained 70% (Fe,Mn)WO_4 at room temperature was finally investigated by X-ray diffraction. Characterization of the sample are structural analysis by XRD as shown in

Figure.1. The crystal structure was observed orthorhombic. The average lattice parameters were observed a = 4.7535A˙, b = 5.6818A˙ and c = 5.0120. Thermal analysis by TG-DTA as shown in Figure.2, the weight loss was observed 4.599% from TGA curve and the oxidation process occurred from the exothermic peak at 464.56 °C from DTA . The phase changes of 70% $(Fe,Mn)WO_4$ powder were investigated at 450°C and 500°C in three hour. The phase changes of 70% $(Fe,Mn)WO_4$ samples were observed from orthorhombic structure to monoclinic structure at 450°C and 500 °C in three hour each as shown in Figure.3 and Figure.4.

X-ray diffraction (XRD) patterns of natural $(Fe,Mn)WO_4$ and annealed $(Fe,Mn)WO_4$ in alkaline media at 300°C as shown in Figure.5 and Figure.6. It was found that the structure of natural $(Fe,Mn)WO_4$ is monoclinic. The average lattice parameters were observed a = 4.7722 Å, b = 5.7218 Å and c = 4.9791 Å. The diffractograms for natural wolframite in hydroxide media show that the most intense and well-defined peaks are attributable to $(Fe,Mn)WO_4$. It was found that the structures of natural wolframite in sodium hydroxide solution was also monoclinic. The average lattice constants are calculated to be a = 4.7529 Å, b = 5.6919 Å and c = 5.0209 Å from the refinement of the XRD data. The other secondary peaks is gradually reduced by mixing with sodium hydroxide solution. This sample was also heated in 450°C, 500°C and 600°C are displayed in Figure.7, Figure.8 and Figure.9. The crystal structure of this sample in 450°C was observed orthorhombic and it was observed that the primary peak is sodium molybdenum oxide hydrate and the secondary peak is $(Fe,Mn)WO_4$. The secondary peak of $(Fe,Mn)WO_4$ is reduced at 500°C in hydroxide media but the crystal structure was also observed orthorhombic. The $(Fe,Mn)WO_4$ peaks were disappeared and sodium molybdenum oxide hydrate peaks were observed in 600°C but the crystal structure was also observed orthorhombic.

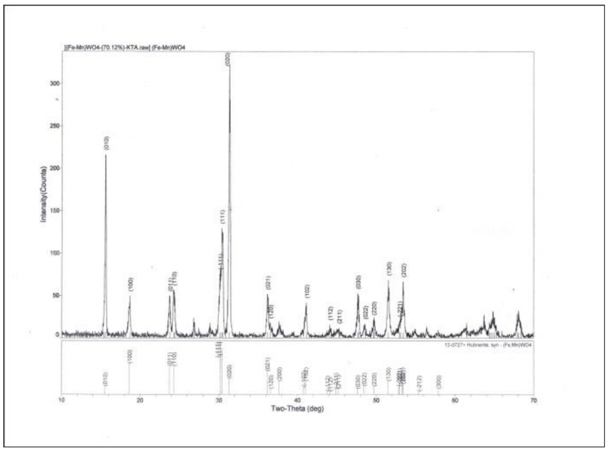

Figure 1 XRD pattern of wolframite 70% $(Fe,Mn)WO_4$

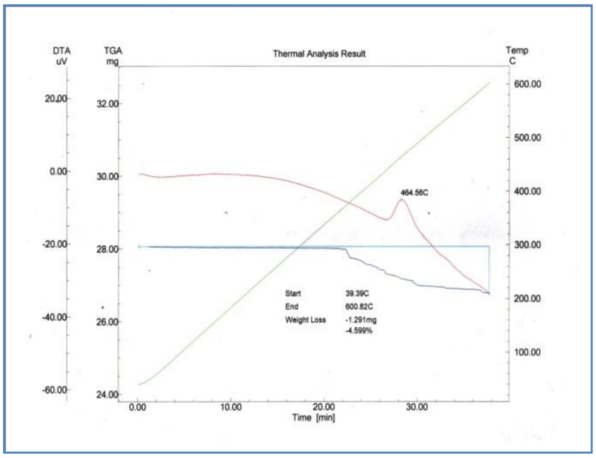

Figure 2 TGA and TD curve of the $(Fe,Mn)WO_4$

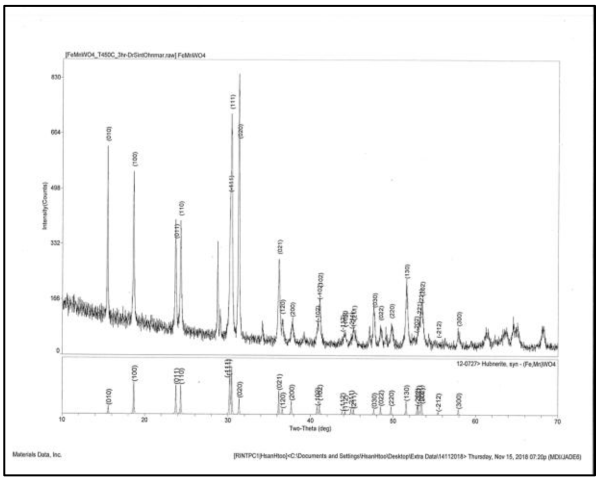

Figure 3 XRD pattern of 70% (Fe,Mn)WO_4 at 450 °C in three hours

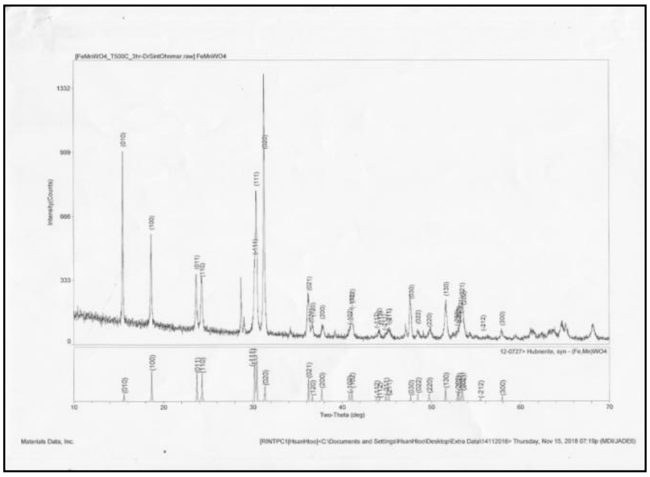

Figure 4 XRD pattern of 70% (Fe,Mn)WO_4 at 500 °C in three hours

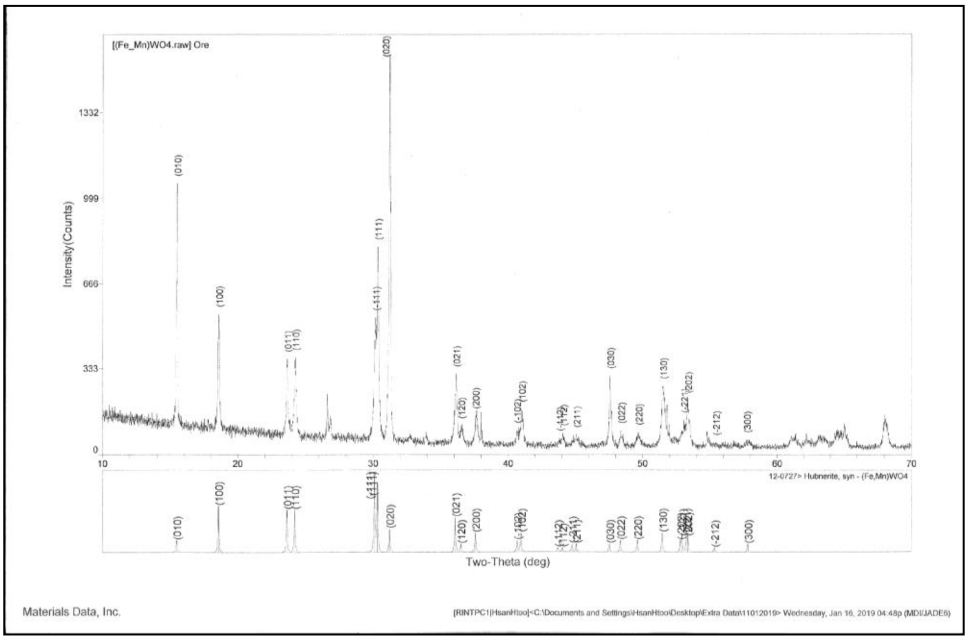

Figure 5 XRD pattern of natural wolframite powder samples

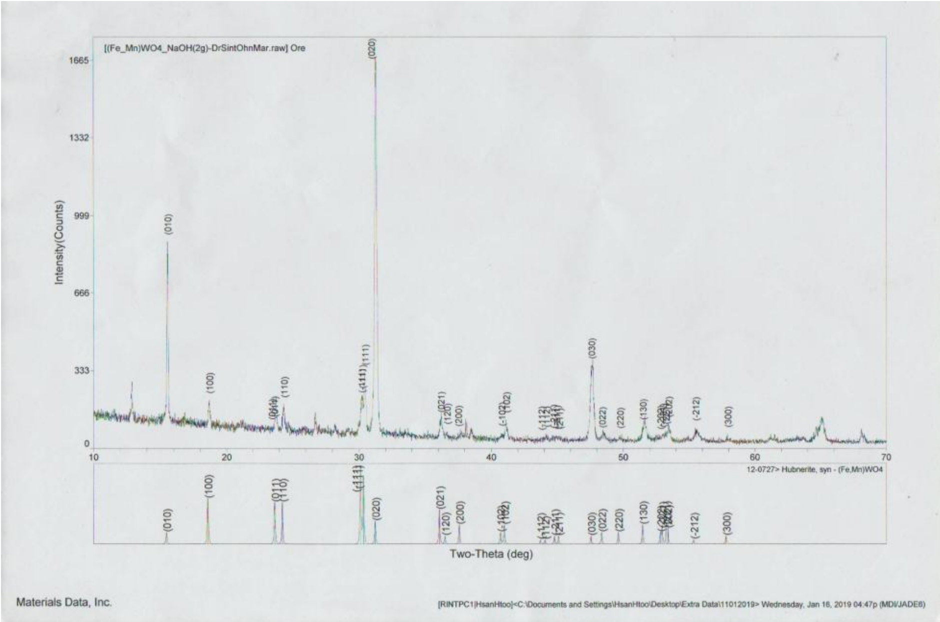

Figure 6 XRD pattern of natural wolframite powder samples with sodium hydroxide solution

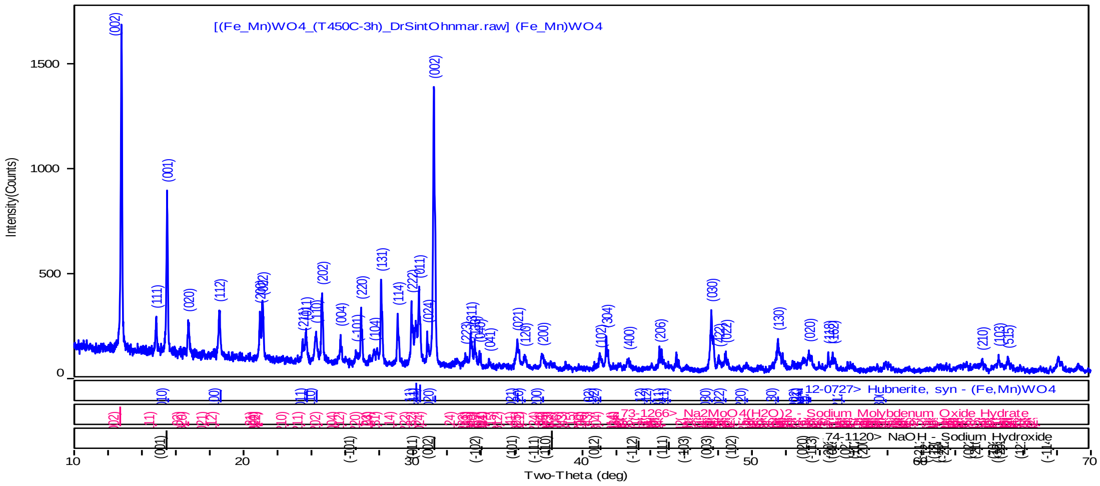

Figure 7 XRD pattern of natural wolframite powder samples with sodium hydroxide solution at 450 °C in one hours

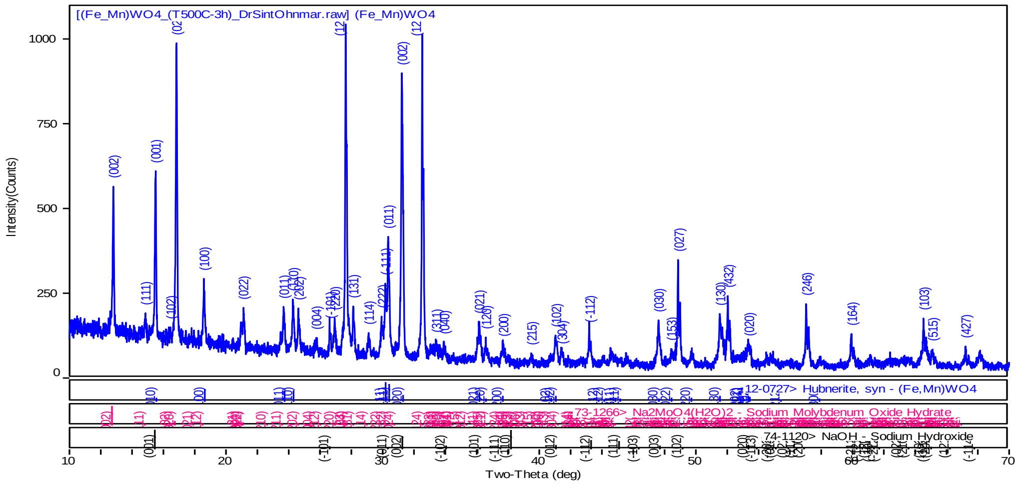

Figure 8 XRD pattern of natural wolframite powder samples with sodium hydroxide solution at 500 °C in one hours

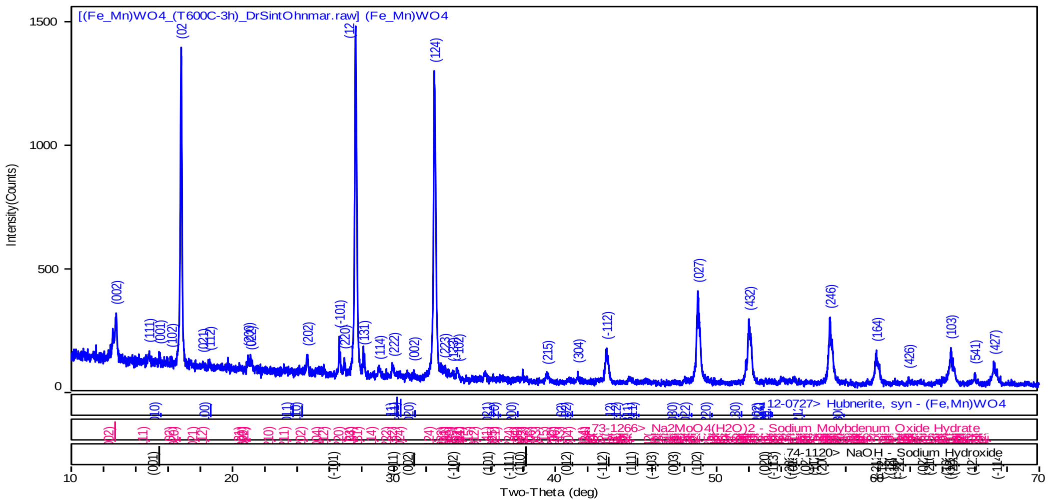

Figure 9 XRD pattern of natural wolframite powder samples with sodium hydroxide solution at 600 °C in one hours

Conclusion

The mineral wolframite is a principal ore of tungsten. It is a general term for iron and manganese tungstates where the iron/manganese ratio can vary. A mineral with more than 80% $FeWO_4$ is called feberite and a mineral with more than 80% $MnWO_4$ is called hubnerite. The primary objective of this work, the characterization of an 70% wolframite which was prepared by magnetic separation method and ore bearing wolfrmite mineral from Pharchaung mine, Tanintharyi region. The XRD phase patterns confirmed the availability of minerals such as Cassiterite, Hubnerite and Feberite. The crystal structure of 70% wolframite from magnetic separation method was observed orthorhombic and structure of the ore bearing wolfrmite mineral observed monoclinic. The mineral confirmed from this mine was hubnerite.

The weight loss of wolframite was observed 4.599% from TGA curve and the oxidation process occurred from the exothermic peak at 464.56 °C from DTA . The phase changes of 70%

$(Fe,Mn)WO_4$ samples were observed from orthorhombic structure to monoclinic structure at 450°C and 500 °C in three hour each but the mineral confirmed hubnerite.

Natural ore wolframite was submitted to chemical and thermal experiments. X-ray diffraction (XRD) patterns of natural $(Fe,Mn)WO_4$ in alkaline media at 300°C in one hour was also observed monoclinic and hubnerite. The structures were stability in alkaline media. In annealing process this sample, the mineral was dominated by sodium molybdenum oxide hydrate than hubnerite at 450°C and 500 °C. The sodium molybdenum oxide hydrate observed at 600 °C in this sample but the structures were changed from monoclinic to orthorhombic in 450°C, 500°C and 600 °C.

Hubnerite is primarily used as a source of tungsten. Tungsten is used to harden metal in the manufacture of high-speed tools. Sodium molybdate is a crystalline powder essential for the metabolism and development of plants and animals as a cofactor for enzymes.

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