Structural Characterization and Magnetization Properties of Tungsten Bearing Ores (Wolframite) Collected From Harmyingyi Mine (Tanintharyi Region)

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ABSTRACT

The tungsten concentrates received from the mining operations are refined in a combination of metallurgical process either to tungsten powders or tungsten carbide powders. The samples were prepared with thermally treated in electrical furnace at temperature of 150°C, 300°C, 500°C, and 700°C, each of 1 hour. Trace element compositions of ores were analyzed by Energy Dispersive X-rays Fluorescence (EDXRF) spectroscopy and the structural properties of these minerals were determined by X-rays Diffraction (XRD) and Scanning electron microscope (SEM). The magnetic properties of these samples were identified with Permagraph L apparatus.

Keywords: Tungsten, Wolframite, X-ray diffraction, SEM and Permagraph L.

Introduction

Tungsten is a metal of high economic importance with unique properties and plays a key role in many technical systems. The element has in its pure form unique properties such as high hardness, good thermal and electrical conductivity as well as the highest melting point of all metals. Accordingly it is used in many different applications where good wear resistance and/or high temperature stability are required. Tungsten is, similar to other refractory metals, brittle at room temperature, which is not only a major concern in many applications but also during the fabrication as it makes mechanical processing more difficult.

Major applications for tungsten are in cutting tools in the form of tungsten carbide, in the production of high speed steel, as an alloying component and as filaments in light bulbs. The most important tungsten minerals are the wolframite solid solution series [$(Fe,Mn)WO_4$] and scheelite ($CaWO_4$). An empirical formula of wolframite is $(Fe,Mn)WO_4$, an intermediate between two end-members: ferberite ($FeWO_4$) and huebnerite ($MnWO_4$).

In general, the formation of tungsten deposits is related to late-stage, highly evolved, granitic intrusions in orogenic belts. Tungsten is enriched by fractional crystallization in the residual melts and is subsequently concentrated in hydrothermal fluids. Tungsten deposits are generally formed by these fundamental processes, although there are several subdivisions of ore deposit types.

In this present work, the wolframite raw mineral was obtained from Harminegyi mine, Tanintharyi region. It was dry-ground in a porcelain mill, and then screened by a sieve. The powder samples were analyzed with X-ray diffraction (XRD) for confirming the present of phases and identifying their structural characterizations. EDXRF spectroscopy shows the element concentrations in these samples and SEM images indicate the formation of group structure and grain boundary. Their magnetization and demagnetization properties of these samples were specified by Permagraph L Technique.

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Materials and Method

Sample Preparation

Rock samples were purchased from Wolframite Harmyingyi mine, Tanitharyi region. Firstly, tungsten-ores alloys were divided and collected from rock sample according to their raw group formations (colour). The obtained raw samples were grounded with agate mortar and heated at 150°C, 300°C, 500°C and 700°C in furnace 1 hr each and slightly cold down to room temperature. The samples were grounded again and again to form homogeneous powder. The powder samples were then added into the internal mixer 3 min after the blending started and allowed to mix for 4 min and were charged into the internal mixer. The powder samples were prepared with XRD holder to determine the structure and lattice parameter.

Once a homogeneous mixture is assumed after 13 min, the samples were pressed by hydraulic press machine into a measurement cell (1.3 cm diameter). The pellet samples were cured in a mold with a spacer approximately 0.5 mm thick and 1.3 cm in diameter by applying pressure of 10 tons. These pellets were taken out and calcined in a furnace at 1100°C for 4 hours. The obtained pellet samples were brownish colour and brittle. The determination of element concentrations, surface morphology and magnetic properties with EDXRF, SEM and Permagraph L apparatus were done in these pellet samples.

Results and discussion

X – ray Powder Diffraction (XRD)

Analysis of X-ray diffraction was performed on the wolframite raw material. The sample was placed in a lucite holder on the goinometer of the XRD-6100 powder diffractometer. The diffraction beam monochromator operated at 20 KVA with step size of 0.02° for 120 minutes to create x- ray patterns with enough intensity to produce lines to identify minerals at the 2θ angles (10° – 80°). Scanning rate was 0.75 degree per minute. Minerals were identified using the ICDD software of the Joint Committee on Powder Diffraction Standard (JCPDS). It was found that the powders were in tetragonal structure having p42/m n m space group. The average lattice constants are calculated to be a = 4.737Å and c = 3.185 Å from the refinement of the XRD data. The prominent peaks in the plot are indexed to various (hkl) planes of Hubnerite ($Fe,MnWO_4$). The secondary peaks were observed in the form of Ferberite ($Fe\ WO_4$) and Dolomite (Ca,MgC_2O_6). The sample calcined at 500°C and 700°C are having stronger peak of (211) plane compared with other samples, but there have more other planes and impurities. The main minerals found in the sample were hubnerite with ICDD card numbers 96-900-8132, the peak analysis using the Guassian curve fitting gave the percent area integration of the various phases which correlated to the quantity of the various minerals in the ore. The XRD pattern of wolframite raw powder (unprepared) was shown in Figure 1(a) and the XRD pattern of samples with various annealing temperature were shown in Figure 1(b).

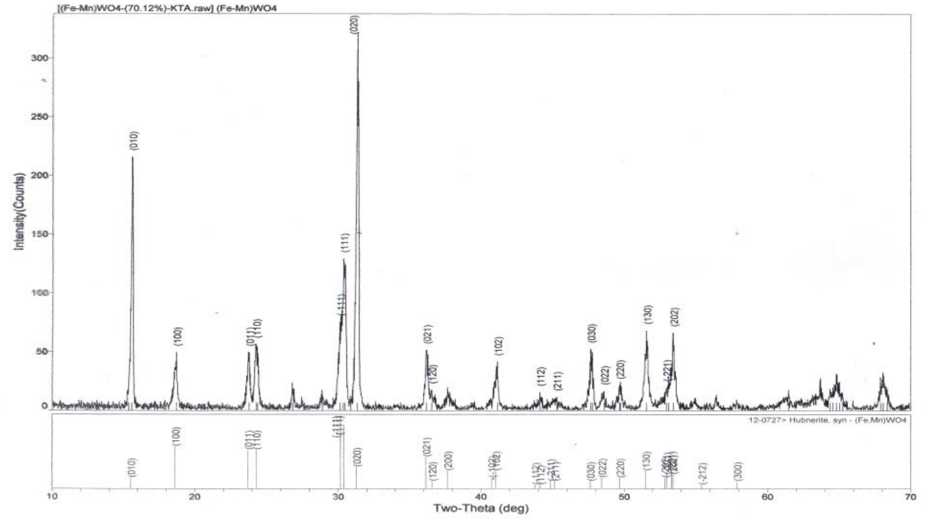


Figure 1(a) : The XRD pattern of wolframite raw mineral.

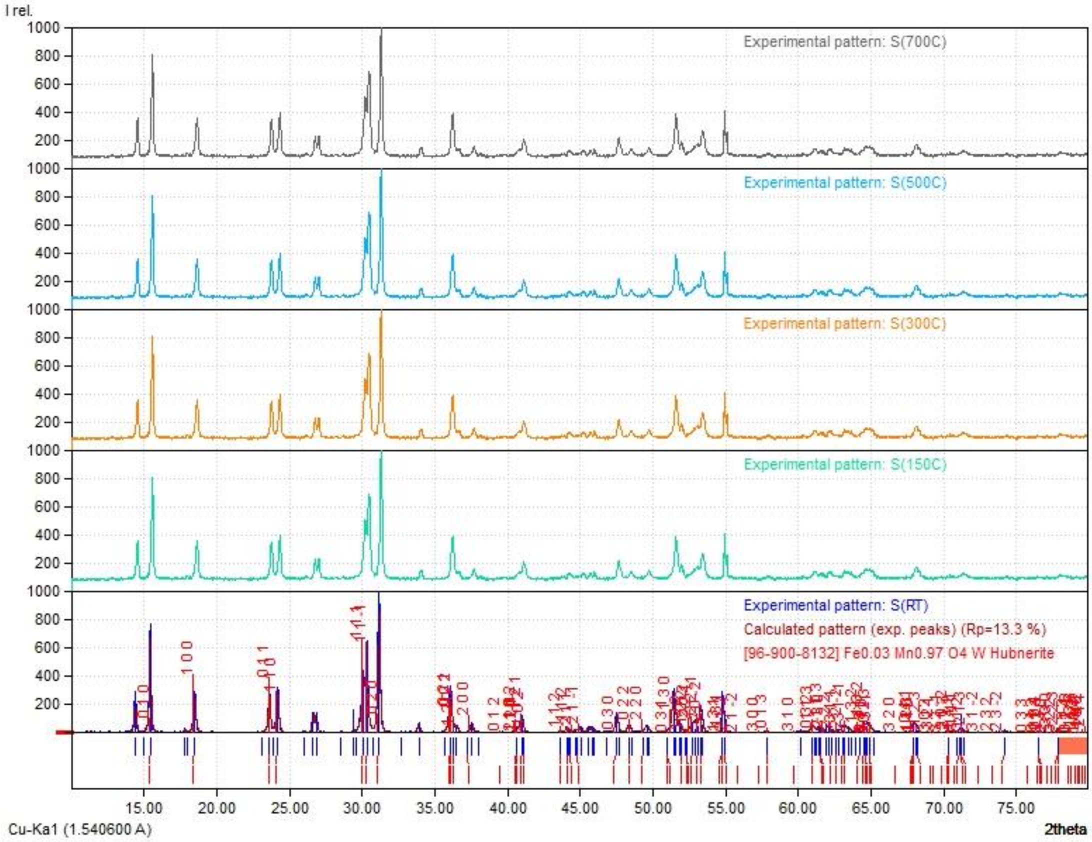
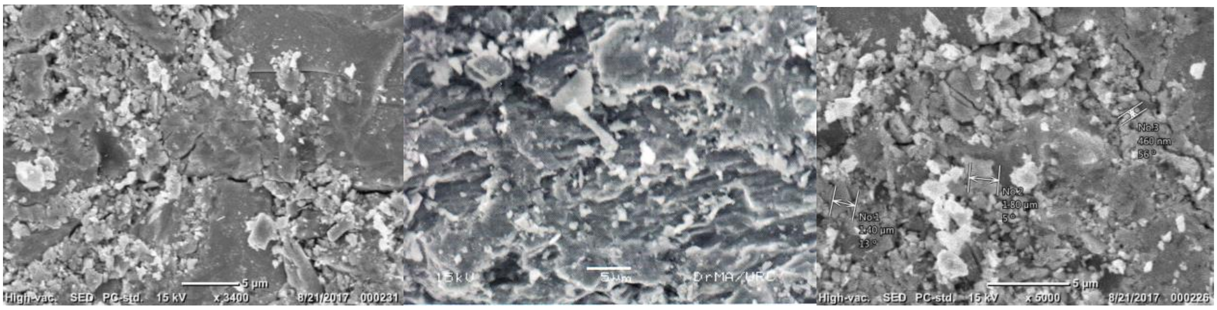


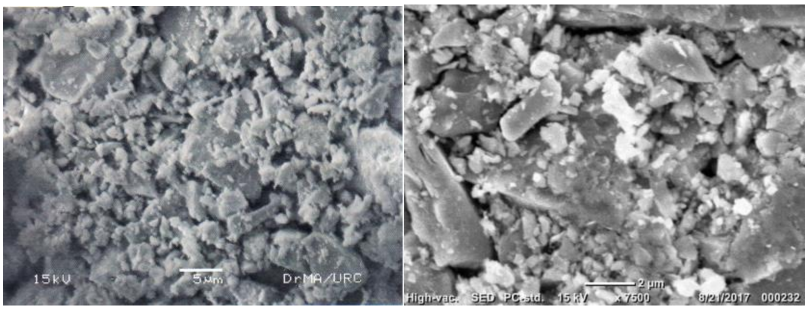
Figure 1(b) : The XRD pattern of wolframite powder with annealing 150°C, 300°C, 500°C and 700°C.

Scanning Electron Microscopy (SEM)

The morphology of the wolframite ore was analyzed in a JEOL JSM-6400 scanning electron microscope at accelerating voltage of 20KVA, real time of 21-36 and live time of 60 seconds. Figure shows scanning electron micrographs of the milled, compacted and calcined samples. There were two different types of morphological feathers along with voids are visible. The unreacted $MnWO_4$ grains which exhibit flowery feathers depict breaking up morphology, evenly distributed on the whole pellet surface. The increase in the furnace temperature enhances the rate of reaction and the images were formed faceted particles and agglomerated rods. The SEM images of these samples were shown in figure 2(a-e).



(a) (b) (c)



(d) (e)

Figure 2 : The SEM image of wolframite mineral annealing with (a) room temperature (b) 150°C (c) 300°C (d) 500°C (e) 700°C.

Energy Dispersive X-ray Fluorescence Analysis (EDXRF)

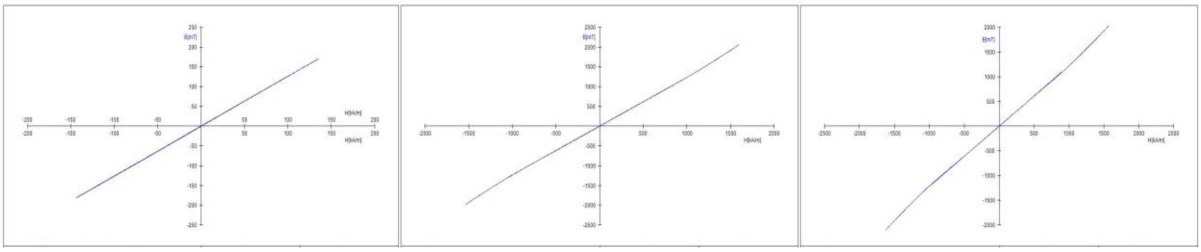
The EDXRF result of the wolframite mineral was shown in table 1. From these results, the main concentration was observed 64.313 % as tungsten (W) and Manganese (Mn), tin (Sn), iron (Fe) were as the major concentrations. The other S, Bi, Pb, Se, Cu, Ca, Nb and Mo were found as the minor elements. Their concentrations were be slightly changed in the range of calcinations temperature 150°C and 300°C. For the calcinations temperature up to 700°C, the percentage of tungsten (W) was increased and minor concentrations were decreased.

Table 1 : The elemental concentrations of wolframite samples

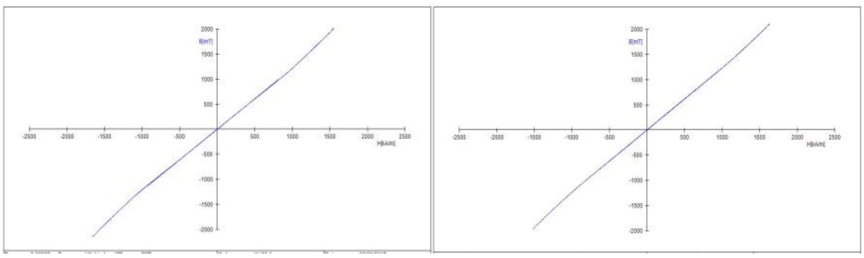
Elements	Concentration (%)				
	room temperature	Calcinations for 1 hour			
		150°C	300°C	500°C	700°C
Tungsten (W)	64.313	64.674	65.001	68.112	72.021
Manganese (Mn)	11.387	11.552	11.984	10.486	9.662
Tin (Sn)	9.217	8.350	8.205	7.221	5.534
Iron (Fe)	7.514	7.226	6.711	6.414	5.013

Magnetic Properties (Permagraph L Apparatus)

The process of magnetization consists of the movement of domain walls so that favorably oriented domains grow fast and the unfavorably oriented ones shrink. If the resistance to the movement of the domain walls is small, the coercive force is small and it is easy to demagnetize the material. Such materials are called soft materials. If the resistance to the movement of the domain walls is large, the coercive force is large and the material is called a hard material. The magnetization and demagnetization properties of wolframite mineral were shown in Figure 3 (a-e). From these results, the remanent value and maximum energy product of these samples were increased with increasing calcinations temperature. Table 2 shows the value of magnetic parameter of wolframite samples.



(a) (b) (c)



(d) (e)

Figure 3 : The Magnetization and demagnetization properties of wolframite mineral calcinations with (a) room temperature (b) at 150°C (c) at 300°C (d) 500°C (e) 700°C.

Table 2 : The magnetic parameter of wolframite mineral.

Magnetic parameters	room temperature	Calcination for 1 hour			
		150°C	300°C	500°C	700°C
Remanence (B_r) (T)	0.000489	0.00195	0.00349	0.00391	0.00996
Normal coercivity (H_{CB}) (kAm^{-1})	0.382	1.57	2.89	3.2	4.57
Intrinsic coercivity (H_{CJ}) (kAm^{-1})	2.5	12.08	37.6	71.6	120.8
Maximum energy product $(BH)_{max}$ (kJm^{-3})	0.00005	0.00075	0.00090	0.00320	0.00750

CONCLUSION

The primary objective of this work was the characterization of an ore bearing wolfrmite mineral from Harmyingyi mine, Tanintharyi region. The mineralogical studies carried out with SEM point imaging showed the presence of different aggregates of minerals. The chemical elemental composition determined by EDXRF were W, Mn, Sn, Fe, S, Bi, Pb, Se, Cu, Ca, Nb and Mo. XRD phase patterns confirmed the availability of minerals such as Hubnerite, Ferberite, Dolomite, cassiterite, manganocolumbite, and quartz . The Permagraph L apparatys conforms that by suitable heat treatments, high values of (B-H), coercivity and remanence can be achieved in these materials.

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