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STUDY ON TEMPERATURE VARIATION OF DENSITIES OF ANTIMONY AND BISMUTH USING GAMMA RAY ATTENUATION TECHNIQUE

Strictly as per the compliance and regulations of :

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Abstract - The densitometer was designed and fabricated with the underlying principle of gamma (γ) ray attenuation produced on passing a collimated beam of monochromatic gamma radiation through any material. After standardization, the gamma ray attenuation coefficient **(μ)** was calculated to determine changes in densities as a function of temperature of Sb and Bi in solid phase. The density of **Sb** and **Bi** at room temperature are **6.697×10^{3} Kgm $^{-3}$** and **9.79×10^{3} Kgm $^{-3}$**, and their melting points are **903.78 K** and **544.7 K** respectively. The measurements were conducted below melting point. The experimental results are in reasonable agreement with published data and may be used as reference data on variation of densities at various temperatures of solids.

I. Introduction

Metals like Sb, Bi have wide range of applications in various fields. Sb is used as flame retardant while its alloys for batteries, bearings and solders. It is also being used in the semiconductor industry, medical and biological fields. Bi is a soft silvery metal with bright surface and a yellowish or pinkish tinge. The metal breaks easily and it expands as it solidifies. This property makes Bi useful for producing 'type metal'. Many alloys of Bi have melting point as low as 343 K, which are used in fire sprinkler systems, fuel tank safety plugs, solders etc,.

The physical properties and structure of materials change with temperature. Thus, it is interesting to researchers in Engineering and Science to study the physical properties as a function of temperature. Density is a basic parameter which decides nature and behavior of materials. The density and thermal expansion values of materials are useful for a variety of scientific and technological applications. Besides, other thermo-physical properties such as viscosity, surface tension, thermal conductivity, etc., can also be determined. The gamma attenuation technique [6] for density determination of materials have several advantages at high temperatures over other methods. The probe-sample compatibility problem, formation of oxide surface films etc. are totally eliminated; G.Dillon,et al. [1], F.E.Levert, et al. [2], Doge [3] and W.Drotning [4 ,5].

II. Experimental Details

a) A brief outline

Gamma Ray Attenuation technique is a non contact – non invasive method that can be used for measuring Thermal Expansion and Density of materials in solid state as well as in molten state and through melting Temperature as well. The Gamma Ray Attenuation Technique used for determination of thermo-physical properties of metals offers several advantages over other methods. This method is a noninvasive one utilizing the gamma beam as a probe which is neither in Physical nor in Thermal contact with the sample. This technique is particularly advantageous at High temperatures as Thermal loses are minimized. This technique also ensures the elimination of incompatibility of sample and probe materials. In measurement of Density by this method, only the solid or molten material of the samples are involved, eliminating the free liquid surface which has no role to play what so ever. Thus a number of problems encountered during the measurements, due to viscosity effects, sample vaporization, surface tension effects, formation of oxide films on surface, etc., by other methods, and their corresponding corrections in calculations are safely avoided.

A Gamma ray densitometer operates on the basic principle of Gamma ray attenuation caused when the gamma ray beam is interrupted on its path to strike a counting system. The basic design requirement comprises of a Gamma radiation source of detectable strength (intensity), a lead vault to house the source, lead collimators to allow a collimated beam to pass to the counting system, a counting system with lead shielding to avoid unwanted radiation.

In this work we conducted study on the densities of Sb and Bi metal samples as a function of temperature. For this purpose we have added a furnace in the path of gamma beam in which the sample is placed and its temperature is incremented or decremented in a standard linear pattern. The measurements are taken at different temperatures to study the variation of. Since the melting point of lead is

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nearly 401 K, lead collimators have been avoided inside and in the close vicinity of the furnace, as we have designed the furnace to reach to a temperature of around 1300 K. Instead, stainless steel collimators have been employed replacing the lead collimators near high temperature regions in the entire setup.

The gamma ray densitometer was designed and fabricated in our laboratory. The cross sectional view is shown in Figure 1. In this paper, an experimental apparatus using the gamma attenuation method with the furnace temperature variation of up to 1300K is described and temperature variation of densities of Sb and Bi were studied. [4, 5].

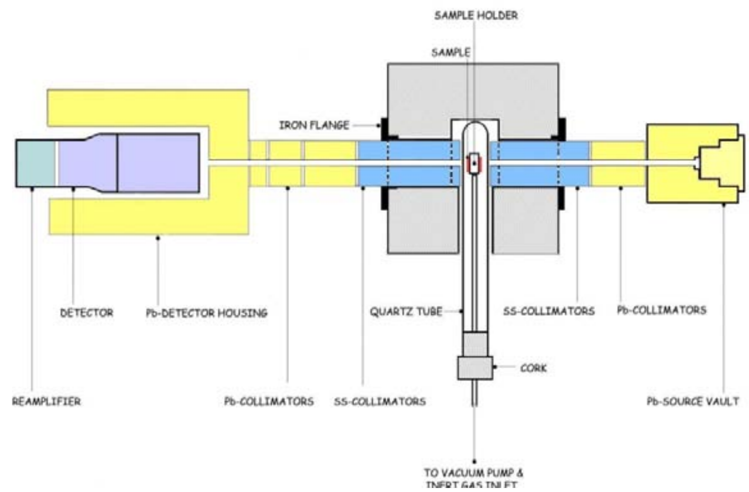

Figure.1

The source vault is made in Lead - 30cm length x 27.5cm diameter, in which the source 137Cs is housed and a collimated gamma ray beam is obtained by using collimators of Lead (5cm, 7.5cm, 10cm length and 6cm diameter) and stainless steel (10cm, 15cm length and 6cm diameter) with an aperture of 6mm diameter. Detector housing made in lead is 30cm in length x 20cm in diameter.

The programmable temperature controlled furnace has been specially designed to fit perfectly into the setup and can reach temperatures up to 1300 K. The instrumentation for the furnace has been designed such that, feedback and control of the furnace can be handled directly from the control panel placed remotely. The metal samples are placed inside the furnace in the path of gamma beam mounted on a sample holder. The sample holder is made up of a flat stainless steel strip bent in the form of a circle and mounted on one end of a stainless steel tube. The sample holder tube passing through a cork is slid into the quartz tube. The tube also assists in evacuation of the quartz tube by employing a vacuum pump and provides inert atmosphere by introducing argon gas. The quartz tube with the sample holder in place, is passed in to the furnace through an orifice at the bottom, drilled through the insulation and outer body of the furnace. A separate wire type thermocouple in perfect physical contact with the sample indicates the exact sample temperature. To detect and analyze gamma ray spectrum NETS-3M/U IK Multi Channel Analyzer in conjunction with a NaI(Tl) detector along with HV supply and preamplifier is used. The gamma radiation source 137 Cs with energy 661.6 Mev and 30 millicurie strength was used.

The PTC furnace was programmed in such a manner that the temperature is incremented by 50K in every step from room temperature and stabilizes at that point for a certain length of time. At each temperature, γ - counts with sample $[I]$ and without sample $[I_o]$ were detected and recorded using a multi-channel analyzer. This process was repeated at every temperature for at least nine times. The counts were recorded while heating and cooling the sample. The difference in the reading was negligible and hence the final readings were recorded while cooling the sample. This procedure was repeated until the desired temperature range was covered in each case.

The setup was standardized using aluminum solid sample as sample and temperature variation of density was found which was in reasonable good agreement with the reported data.

III. Analysis of Date

In the present work, the γ - attenuation technique has been applied for the measurement of density of solid materials. In addition to density, this technique allows measurement of both linear thermal expansion in the solid state and the volumetric thermal expansion in the liquid state without any change in experimental conditions. The data on all the samples have been analyzed using the analytical method given by Drotning.

The basic equation which defines the γ-attenuation for mono-energetic gamma rays and a narrow beam geometry. is

$$I(T) = I_O(T)\exp[-\mu\rho(T)l(T)]$$

where $I_o(T)$ and $I(T)$ are the gamma intensities before and after passing through the sample material, $\rho(T)$ is the sample density, l is the sample length along the γ - ray path, T is the temperature of the sample and μ is the mass attenuation coefficient of the sample material. The mass attenuation coefficient is independent of the physical condition of the sample and hence is independent of temperature. The temperature dependence of I_o includes temperature induced attenuation changes in the furnace, chamber materials and quartz tube, as well as size changes in the collimation.

IV. Results and Discussion

The temperature dependence of density of Sb and Bi from room temperature to close to their annealing temperatures are plotted. Results are in agreement with published data [7]

a) Variation of Density of Sb.

Fine powder of Sb is made into a pellet of diameter 2cm with a die set by applying 3000psi pressure. The Pellet of length of 1.655 cm. is placed in the sample holder and sintered at 823 K temperature for four hours to form into a solid. A collimated beam of gamma radiation is passed through the sample at different temperatures in decreasing order with intervals of 50 K. The counts are recorded before the sample is introduced into the furnace (I_0) and after the sample is introduced into the furnace (I), at each value of temperature. The linear attenuation coefficient (μ) is determined and the density at each temperature is calculated. The temperature variation of density for Sb, is plotted in Fig. 2. The values of I_0, I, I_0 / I and ρ are tabulated in Table 1.

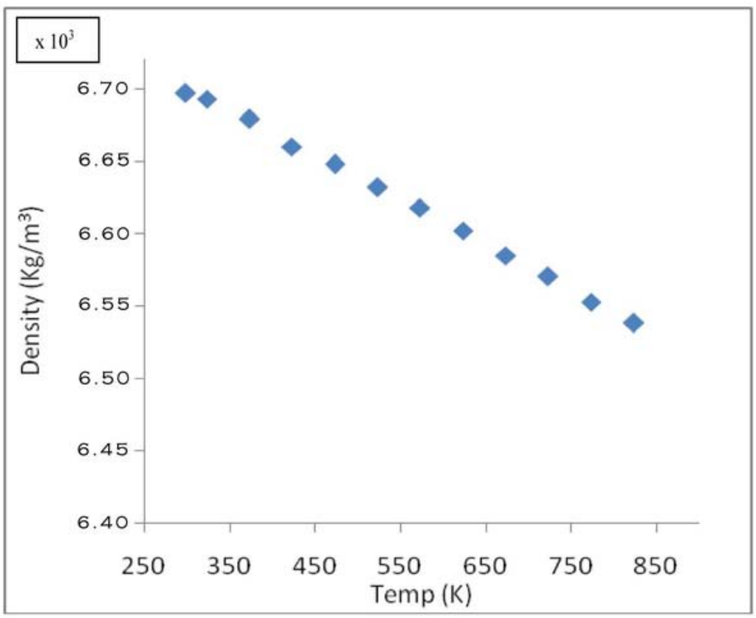

Figure.2 : Temperature variation of density of Sb.

Table 1 : Temperature variation of density of Sb.

Temperature (K)	I_0	I	I_0/I	ρ (Kg/m^{3})
298	42972	24581	1.748159	6697.0000
323	42970	24584	1.747914	6693.4771
373	42580	24382	1.746417	6679.5327
423	42404	24311	1.744218	6660.7820
473	41779	23971	1.742913	6648.1642
523	40858	23467	1.741108	6632.1255
573	41717	23981	1.739617	6618.2465
623	40742	23443	1.737914	6602.9201
673	40209	23164	1.735819	6584.8984
723	38711	22322	1.734241	6570.4395
773	37551	21678	1.732194	6552.7613
823	32188	18599	1.730604	6538.2301

b) Variation of Density of Bi.

Fine powder of Bi is made into a pellet of diameter 2cm with a die set by applying 2500psi pressure. The Pellet of length of 1.315cm. is placed in the sample holder and sintered at 473 K temperature for four hours to form into a solid. A collimated beam of gamma radiation is passed through the sample at different temperatures in decreasing order with intervals of 25 K. The counts are recorded before the sample is introduced into the furnace (I_0) and after the sample is introduced into the furnace (I), at each value of temperature. The linear attenuation coefficient (μ) is determined and the density at each temperature is calculated. The density variation with temperature is plotted in Figure 3. The density variation with temperature is plotted in Fig. 2. The values of I_0 , I , I_0 / I and ρ are tabulated in Table 2.

Figure.3 : Temperature variation of density of Bi.

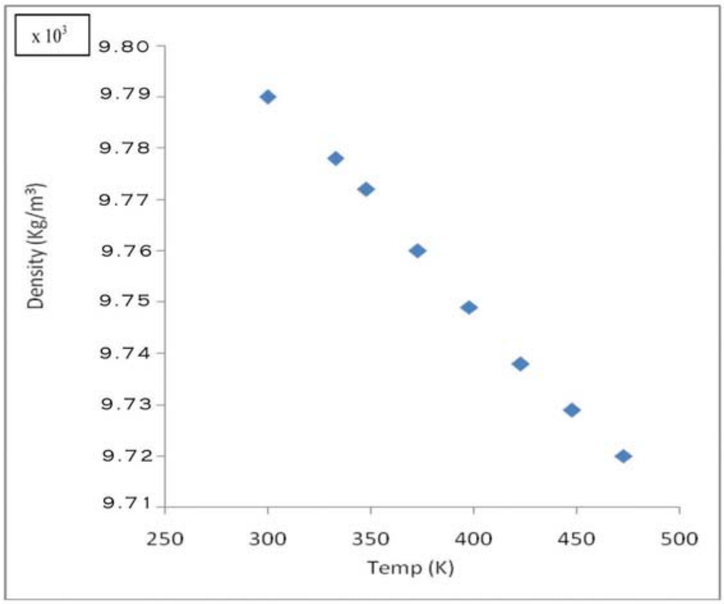

Table 2 : Temperature variation of density of Bi.

Temperature (K)	I_0	I	I_0 / I	ρ (Kg/m^{3})
300	38094	13531	2.815276478	9790.0
333	37956	13493	2.812860248	9777.6
348	37769	13432	2.811805676	9772.0
373	37621	13392	2.809099507	9759.7
398	37493	13357	2.806920933	9749.1
423	37429	13346	2.804568993	9737.9
448	37386	13338	2.802928950	9729.1
473	37324	13324	2.801104051	9719.7

V. Conclusions

From the calculated values, the density of Sb and Bi as a function of temperature by gamma ray attenuation method, it is noticed that the density is decreasing due to increase in linear expansion of material with the increase in temperature. The density of Sb has been ranging in between 6.697x 103 Kgm^{-3} and 6.538x 103 Kgm^{-3} in the temperatures 298 K and 823 K . The density of Bi has been ranging in between 9.79x 103 Kgm^{-3} and 9.719x 103 Kgm^{-3} in the temperature range 300 K and 473 K The results obtained are in agreement with already published data [7] calculated by other methods.

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