

X- Ray mass attenuation coefficients for mixture of some 3d elements $Fe_{100-x} - Al_x$ and $Cu_{100-x} - Al_x$

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Abstract: In this paper we study the mass attenuation coefficients for ($Fe_{100-x} Al_x$, $Cu_{100-x} Al_x$) Mixtures with different weight ratios. These mixtures were prepared by mixing the Fe and Cu With different concentration of Al ($x = 10-50$) using ball milling technique. The mass attenuation coefficients (μ_m) were measured by self attenuation of X-rays at the energy of 30 keV. The Gamma rays emitted from Am- 241 source were used to excite the mixture samples and emitted K_α and K_β X-ray lines were counted by a Silicon drift (SDD) detector with a resolution of 150 eV at 6.4 keV. The samples have also been characterized by SEM and (EDXRF) measurements. The obtained results were compared with theoretical values and are agreement with theoretical results.

Key words: Mass attenuation coefficient, X-ray fluorescence, 3d transition elements, scanning electronic microscope (SEM), EDAX.

INTRODUCTION

The X-Ray Fluorescence (XRF) become a powerful and versatile technique to analysis and characterization of materials (R. Novelline, 1997) (Chilton *et al.*, 1984). The information gained by experimental values of K_α and K_β X-ray production and cross sections have a wide use and applications (Rene, E. Grieken and Andzerj Markowski, 1993). Accurate values of mass attenuation coefficients are essential when quantifying the interactions of an X-ray beam with matter. They are functions of both the photon energy and the physical properties of the irradiated material, The mass attenuation data used in Bremsstrahlung spectra model, exposure and dose calculations (W. Herman, 1994). The mass attenuation compilations do not however cover every energy value in the energy ranges normally presented. To interpolate values for the mass attenuation coefficients for energies of interest, other than those tabulated, the compiled data are often fitted utilizing least-square techniques and appropriate mathematical expression (S.M. Seltzer., J.H., Hubbell, 1995) (J.H. Hubbell and S.M. Seltzer, 1996). When a beam of x-ray photons passes through attenuated material, each photon in the beam either doesn't react with the medium material at all or it reacts by absorption and scattering reactions. Due to the removal of photons from the beam, it will suffer attenuation, such attenuation may be intensity attenuation or energy attenuation, and such that the intensity or the energy decreases along the path it passes through this medium. Not all vacancies causes the production of characteristic X-ray photons since there is a competing internal rearrangement process known as the Auger effect (I. Rodríguez, Cabo, 2005). The ratio of the number of vacancies resulting in the production of characteristic X-ray photons to the total number of vacancies created in the excitation process is called the fluorescent yield.

Fluorescent yield values are several orders of magnitude less for the very low atomic numbers. In the literature description of mixtures of elements, it is standard practice to assume that the contribution of each element to the attenuation is additive. This assumption yields the well known mixture rule which gives the attenuation coefficient of any substance as the sum of the appropriately weighted contributions from the individual atoms. Mass attenuation and energy absorption coefficients are widely used in the study of interaction of γ -rays with matter. Many measurements have been reported (O. Icelli., S. Erzeneoglu and B. Gurbulak, 2005) (A. Khanna *et al.*, 1996) (M. Ertuğrul, 1997) (I. celli Orhan and Salih Erzeneoğlu, 2004). Further advances on the conventional modes of these techniques especially related to the formation of exciting micro beams (X-ray or ion beams) provided elemental mapping capabilities down to the few parts per million concentration regime, enhancing considerably the applicability of the ion-beam- and X-ray spectrometry-based techniques into the cultural-heritage related studies (K. Janssen's *et al.*, 2000) (A. Von Bohlen *et al.*, 2007) (B. Constantinescu *et al.*, 2008) (N. Grassi *et al.*, 2007) (M.J., Berger and Hubbell, S. H., 1987). In present paper we study the mass attenuation coefficients (μ_m) for mixture some 3 d transition elements ($Fe_{100-x} - Al_x$, $Cu_{100-x} Al_x$) with different weight ratio by self attenuation method using X-ray fluorescence. The sample mixtures were prepared by mixing the Fe and Cu with different weight fraction Al using ball milling technique. The sample has also been characterized by SEM and EDAX.

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2.Theory:

When a beam of monoenergetic photon falls with initial intensity (I_0) on a medium of thickness (dt), the decrease in intensity (dI) due to reaction of photons by scattering and absorption in the medium can be expressed by equation 1(W. R. Berlin., Leo, 1987).

$$I = I_0 e^{-\mu t} \quad (1)$$

Where I is the intensity of the attenuated beam, I_0 is the initial intensity of the beam, t is the thickness of the attenuating material in cm, and μ is the linear attenuation coefficient of the attenuating material, then by dividing μ on density (ρ) we got the mass attenuation (μ_m). The linear attenuation coefficient depends on the material and the energy of the incident x-ray (S. Sewell, 8.13 course reader., 2007). Since all emitted X-ray photons have energies proportional to the differences in the energy states of atomic electrons, the lines from a given element will be characteristic of that element. The relationship between the wavelength of a characteristic X-ray photon and the atomic number Z of the excited element (equation 2) was first established by Moseley (M. A. B. Wiltaker.,1999).

$$1/\lambda = k(Z - \sigma)^2 \quad (2)$$

In which K is a constant that takes on different values for each spectral series, σ is the shielding constant that has a value of just less than unity. The wavelength of the X-ray photon is inversely related to the energy E of the photon according to equation 3.

$$\lambda_0 = \frac{12.4}{E(\text{keV})} \quad (3)$$

Thus the mass attenuation coefficient μ_m is given by :-

$$\mu_m = \sum_i w_i \left(\frac{\mu}{\rho} \right)_i \quad (4)$$

where w_i is the proportion by weight, μ is the linear attenuation coefficient in cm^{-1} , ρ (g/cm^3) is the density of element and μ_m (cm^2/g) is the mass attenuation coefficient of constituent element i of the compound. The mixture rule is valid when the effects on the atomic wave function of molecular bonding and chemical or crystalline environment are negligible. However, the μ/ρ value for an element in a compound may vary substantially for incident photon energies that lie above the element's absorption edge. The μ_m values are also believed to be affected by the chemical, molecular and thermal environments. These phenomena lead to the deviation of the experimental μ_m value from that of the theoretical value, since the calculation of the theoretical value has been done by considering the cross-section of an isolated atom. This deviation is termed as the breakdown or the no validity of the mixture rule.

3.Experimental:

3.1 Materials and Sample Preparation:

In the present work the sample mixtures of $\text{Fe}_{100-x} \text{Al}_x$ and $\text{Cu}_{100-x} \text{Al}_x$ were prepared by mixing the Fe and Cu with Al in weight ratio $x = 10\%$, 20% , 30% , 40% and 50% . The high purity (99.9-99.99 %) fine powder of Al, Cu and Fe were obtained from Sigma Aldrich. The mixing has been done using ball milling (Planetary Ball Mill PM100) contained stainless steel container (volume about 75 cm^3) and stainless steel ball having a diameter of 10 mm. We put the sample powder ($\text{Fe}_{100-x} \text{Al}_x$ and $\text{Cu}_{100-x} \text{Al}_x$) separately in ball milling container with ball to powder ratio 10:1 in argon atmosphere. The rotation speed of mill was 250 rpm and milling time, followed by 4hr further grinding with postal mortar.

3.2 Characterization:

The mass attenuation coefficients were determined by measuring of X-rays fluorescent emitted through sample mixture of known thickness. The experimental arrangement is shown in Fig. 1. The sample mixtures were irradiated with 30 keV X-rays obtained from the Am^{241} (40 mCi) annular source. The emitted K-line X-rays fluorescents were collimated by the lead collimator shielded with aluminum and iron to fall on samples. These sample mixture was put in circular ring having various thicknesses (0.5- 1.5) mg/cm^2 . The X-rays fluorescent was recorded with help of silicon drift detector (SDD) connected to the Fast Comtech multi channel analyzer card. The distance between sample to source was 15mm same as the distance between sample to detector with an angle 90° as shown in Fig.1. Total mass attenuation coefficient, μ_m (cm^2/g) of mixtures were calculated by the equations 5.

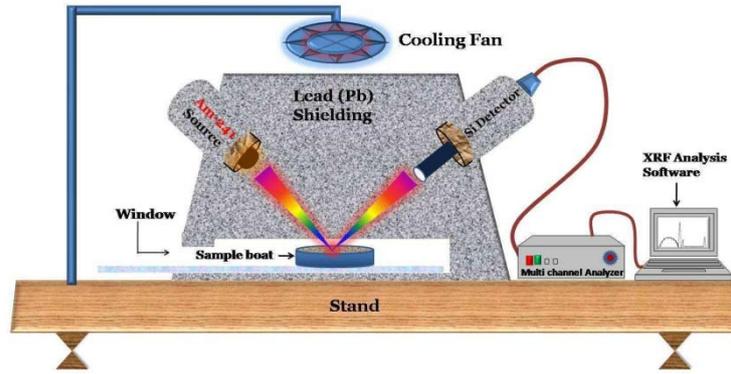


Fig. 1: The experimental setup.

$$\mu_m = \frac{-1}{t} \ln \left(\frac{I}{I_0} \right) \quad (5)$$

Where t (mg/cm²) is the thickness of sample, I and I_0 are area under the peak (K_{α} and K_{β}) for the mixture and pure elements (i.e. Fe, Cu) respectively. The μ_m values obtained experimentally was compared with the theoretical values obtained by Xcom software [16]. The relative difference (RD) between the experimental and theoretical values has been calculated by equation 6.

$$R.D = \frac{(\mu_m)_{exp} - (\mu_m)_{theo}}{(\mu_m)_{theo}} * 100\% \quad (6)$$

The samples have also been characterized using Scanning Electron Microscopy (Zeiss, Germany) and EDAX analysis.

RESULT AND DISCUSSION

4.1 Mass Attenuation Coefficient (μ_m):

The Fig.2 and Fig.3 show the XRF measurements for pure Fe and pure Cu with different weight ratio of Al (10%, 20%, 30%, 40% and 50%). These figures clearly indicate the K_{α} and K_{β} X-ray energy peaks for pure Fe and pure Cu have been appeared at 6.4 KeV and 7.05 and 8.08 KeV and 8.9KeV respectively with higher Intensity. It was observed that for mixture samples intensity of these K_{α} and K_{β} X-ray energy peaks was decreased as the concentration of Al increased.

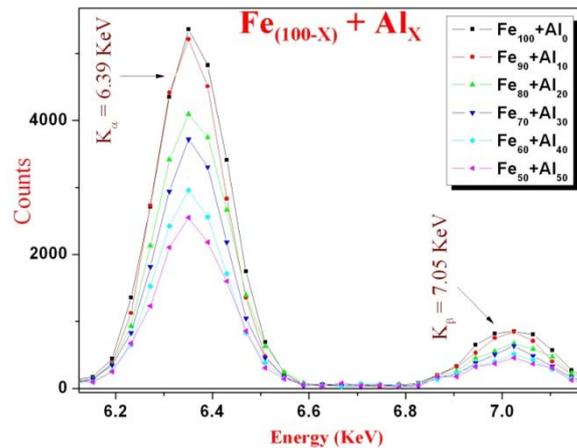


Fig. 2: Shows the X-ray fluorescence plots for $Fe_{100-x}Al_x$ mixtures.

We have calculated the mass attenuation coefficient (μ_m) for these mixtures by Eq. 6 and it was observed that in case of $Fe_{100-x}Al_x$ μ_m is linearly increased as the Al concentration increased in Fe mixture while for $Cu_{100-x}Al_x$ the μ_m decreased as the Al concentration increased. The experimental and theoretical plots of μ_m with Al concentration for Fe and Cu mixtures have been shown in Fig. 5 and Fig. 6 respectively.

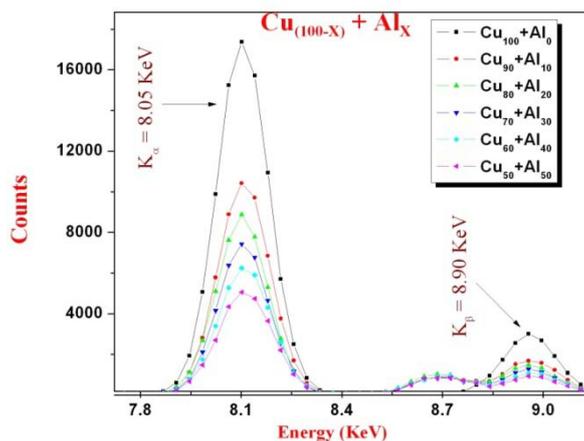


Fig. 3: Shows the X-ray fluorescence plots for Cu_{100-x}Al_x mixtures.

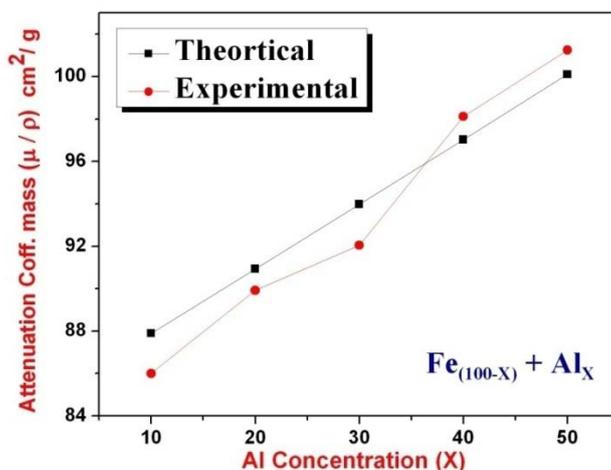


Fig. 4: Shows the experimental and theoretical plot between mass attenuation coefficient (μ_m) and Al concentration for Fe_{100-x}Al_x mixtures.

These measured experimental values of μ_m have been compared with theoretical values by Xcom software and have good agreement. The increased values of μ_m in case of Fe mixture may be attributed to the higher absorption X-ray fluorescence due to presence of Al in mixture. We may assumed that when pure Fe is exposed with incident X-ray then it emits the X-ray fluorescence and hence behaving as source which detected by the detector. As we know that K_α and K_β X-ray energy for Al is less 1.48 KeV and 1.55 KeV respectively and its density is also less in comparison to the pure Fe, therefore in mixture the absorption of X-ray fluorescence emerged from Fe is high therefore been detected which gives the high attenuation then pure Fe. The calculated μ_m for Fe-Al mixture has been shown in table 1.

In case of Cu_{100-x}Al_x mixture we observed just inverse behavior as compared to Fe_{100-x}Al_x mixture that the μ_m is continuously decreased as the Al concentration increased in Cu mixture as shown Fig. 5. It may assumed that in Cu_{100-x}Al_x mixture the Cu is a secondary source of X-ray with higher K_α and K_β X-ray energy at 8.0 KeV and 8.9 KeV (Fig. 3), so this energy is sufficient to interact and eject the electron form inertial shell of Al which gives the higher fluorescence and hence less attenuation. The calculated μ_m for Cu-Al mixture has been shown in table 2.

4.2 Scanning Electron Microscopy (SEM):

The Fig. 6 and Fig. 7 show the SEM images for Fe₅₀Al₅₀ and Cu₅₀Al₅₀ mixtures. These images show that mixtures composed of the uniformly distributed uneven granular structure. The EDAX measurement confirms the elemental composition of Fe₅₀Al₅₀ and Cu₅₀Al₅₀ mixtures.

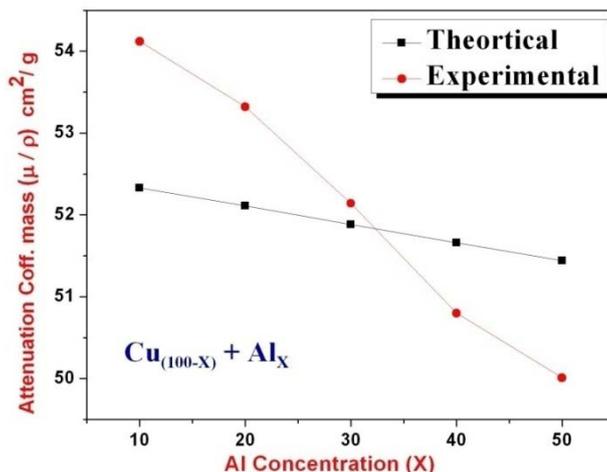


Fig. 5: Shows the experimental and theoretical plot between mass attenuation coefficient (μ_m) and Al concentration for Cu_{100-x}Al_x mixtures.

Table 1: Shows the calculated experimental and theoretical mass attenuation coefficient (μ_m) for Fe_{100-x}Al_x mixtures.

Samples	Weight ratio	μ_m Experimental	μ_m Theoretical	Percentage error
Fe+Al	90%+10%	86	87.89	0.021
Fe+Al	80%+20%	89.92	90.93	0.011
Fe+Al	70%+30%	92.05	93.98	0.02
Fe+Al	60%+40%	98.12	97.02	0.011
Fe+Al	50%+50%	101.25	100.1	0.011

Table 2: Shows the calculated experimental and theoretical mass attenuation coefficient (μ_m) for Cu_{100-x}Al_x mixtures.

Samples	Weight ratio	μ_m Experimental	μ_m Theoretical	Percentage error
Cu+Al	90%+10%	54.12	52.33	0.034
Cu+Al	80%+20%	53.32	52.11	0.023
Cu+Al	70%+30%	52.14	51.88	0.005
Cu+Al	60%+40%	50.25	51.66	0.027
Cu+Al	50%+50%	50.01	51.44	0.027

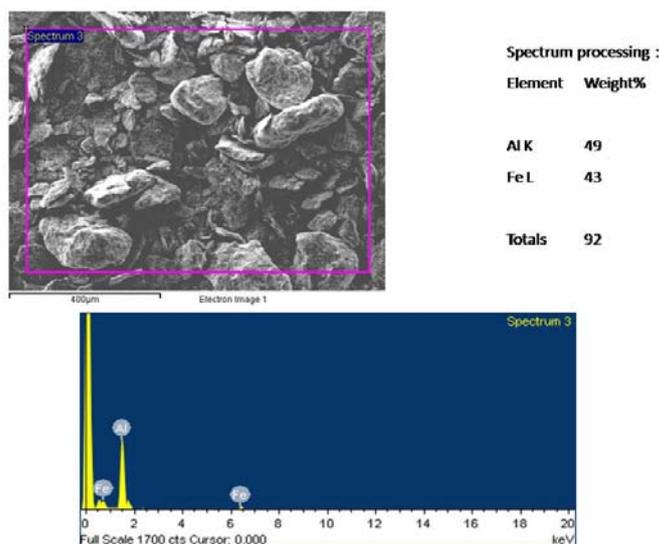


Fig. 6: The SEM image with EDAX analysis for Fe₅₀Al₅₀ mixture.

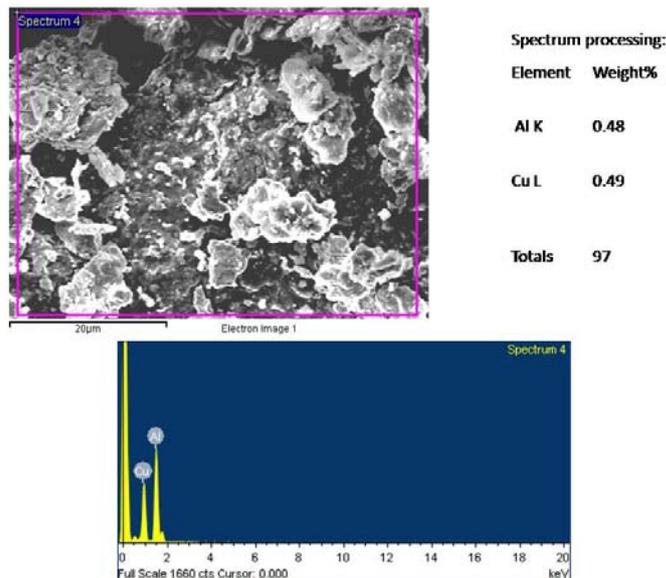


Fig. 7: The SEM image with EDAX analysis for Cu₅₀Al₅₀ mixture.

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