SAC305 SOLDER'S RESPONSE TO GAMMA RADIATION ENVIRONMENT: IMC LAYER, XRD CHARACTERISTICS AND HARDNESS BEHAVIOR

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ABSTRACT

This study explores the impact of radiation dose exposure on the characteristics of solder joints, with a focus on the interplay between the intermetallic compound (IMC) layer thickness, X-ray diffraction (XRD) patterns, and hardness of SAC305 solder. By subjecting solder joints to varying gamma radiation doses, we investigate their responses and draw insightful correlations. Microstructural evolution of Sn-Ag-Cu solder alloys induced by gamma radiation were investigated such as the distribution of primary phase beta Sn (β-Sn) and near eutectic phase, intermetallic compound (IMC) layer thickness and generation of micro-cracks. Characterization and analysis of microstructure were examined via optical microscopy, and X-ray Diffraction analysis. Our findings reveal a direct relationship between radiation dose and IMC layer thickness, as higher doses result in a proportional increase in layer thickness. XRD analysis confirms the presence of characteristic peaks associated with β-Sn, Ag_3Sn, and Cu_6Sn_5 phases, underscoring the structural integrity of the SAC305 solder joints. The mechanical properties of the solder joints exhibit intriguing behaviors with increasing radiation dose. Initial exposure leads to enhanced hardness, suggesting radiation-induced strengthening effects. However, a subsequent decline in hardness occurs as radiation dose is raised up to 500 Gy, possibly indicating a saturation point or radiation-induced softening mechanism. Remarkably, beyond the 500 Gy threshold, hardness begins to rebound as doses reach 5000 Gy and 50000 Gy. This suggests complex interactions between radiation-induced defects and phase transformations within the solder. These findings offer critical insights into the intricate interplay between gamma radiation exposure and the mechanical properties of solder joints. This understanding holds significant implications for designing electronic systems resilient to radiation in high-risk environments.

Keywords: solder joints, intermetallic compounds, X-ray diffraction, hardness, gamma radiation

INTRODUCTION

Nowadays, the reliability and robustness of electronic devices, particularly when operating under extreme conditions such as radiation exposure, have gained significant attention. Electronic devices may encounter radiation across a various of applications and environments, encompassing space exploration, nuclear power plants, medical imaging and treatment, aerospace, and aviation, as well as defence and military applications [1]. The impact of radiation on electronic devices involves both immediate and long-term effects. Radiation exposure can introduce various challenges to the dependable operation of electronic devices, particularly in applications where radiation levels are elevated [2]. Ionizing radiation, such as gamma rays and high-energy particles, can disrupt the atomic and molecular structure of materials within electronic components. This can lead to a range of detrimental consequences, including changes in material properties, degradation of electrical performance, and increased susceptibility to faults and failures [3].

Solder joints are critical components within electronic devices that have a significant impact on their overall reliability and performance. These connections serve as the pathways for electrical signals and power between various components, such as integrated circuits, resistors, capacitors, and connectors [4]. The quality and integrity of solder joints directly influence the device's functionality, longevity, and ability to withstand various operational conditions [5]. Solder joints are typically formed by using solder alloys, which consist of a mixture of metals that melt at relatively low temperatures. Common solder alloys including tin-lead (Sn-Pb), tin-silver-copper (Sn-Ag-Cu), tin-copper (Sn-Cu), tin-silver (Sn-Ag), tin-bismuth (Sn-Bi) and etc [6–8]. The choice of solder alloy depends on factors such as the application, operating conditions, required mechanical and thermal properties, and regulatory considerations. The transition from conventional Sn-Pb solder to Pb-free options like Sn-Ag-Cu is urged by environmental concerns, regulations, and the need for more reliable electronics manufacturing. Sn-Ag-Cu solder offers key benefits over Sn-Pb solder. It eliminates toxic lead, addressing environmental and health risks throughout production and disposal. Sn-Ag-Cu solder boasts improved mechanical and thermal properties, including higher strength, fatigue resistance, and thermal cycling reliability [9].

Based on Yannakopoulos et al. [10], effects of radiation rays on materials can be classified into four main categories. First, radiation rays can lead to the generation of impurities through nuclear transmutation, transforming nuclei into other, potentially radioactive, forms. Second, atomic displacement occurs as radiation displaces atoms from their original positions in the material's structure, resulting in voids or the exchange of atoms within the lattice. Third, ionization takes place, involving the removal of electrons from atoms and the formation of ion pairs, altering the material's electrical properties. Lastly, the release of substantial energy within a confined volume heats up the material, which is particularly significant when the material serves as a radiation shield. These categories illuminate the diverse ways in which radiation interacts with materials, impacting their structural, electrical, and thermal characteristics. Therefore, numerous researchers were conducted the studies to discover the effect of radiation on the properties of materials included metals and alloy which are radiation exposure tendency.

Previous studies have explored the impact of radiation, especially gamma rays, on solder joints, focusing on microstructure and mechanical properties. Wang et al. [11, 12] studied the effect of gamma radiation on Sn-Pb based solders and found that gamma rays caused the formation of micro-voids and micro-crack in the solder joints. Exposure to the gamma rays also degrade the mechanical properties of Sn-Pb based solder joints. They reported that shear force, impact toughness and ductility properties decrease after the long exposure to the radiation. Additionally, Sn-Pb based solder does not exhibit excessively rapid growth of intermetallic compound (IMC) layers. The morphology of the IMC remained in a scallop-like shape, and the thickness of the IMC layer hardly changed [11, 12]. Research conducted by Wen et al. [13]

revealed that gamma irradiation significantly degrades the mechanical properties of AuSn20 solder joints. The solder joint's failure mode shifted from ductile fracture to brittle fracture, and the shear strength of the joint decreased by 37.23% following 1000 hours of γ-ray irradiation. Exposure to gamma radiation leads to the agglomeration of voids, activation of interfacial reactions, and triggers excessive growth of IMC layers, resulting in a substantial reduction in the shear strength of the joints [13]. Lehan et al. [14] investigated the mechanical and microstructure of 96.5Sn3.0Ag0.5Cu (SAC305) solder joints under low doses of gamma radiation. As the radiation dose increased, the SAC305 solder joints became softer and more plastic. Analysis by Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive Spectroscopy (EDS) analysis signified that IMC layer in the tin (Sn) matrix was dominated by Cu_6Sn_5 and Ag_3Sn compound. According to micromechanical investigation, both hardness and reduced modulus of the solder decreased with increasing the radiation dose that was due to microstructure change and the formation of voids [14]. Yusoff et al. [15] reported the impact of gamma radiation on the micromechanical properties of SAC305 solder alloys. The research reveals that exposure to gamma radiation alters the hardness and creep behavior of the solder. Hardening phenomena cause the increasing in hardness values due to the defects induced by gamma irradiation. Additionally, the creep behavior of the solder changes significantly with different gamma doses, showing increased rates at doses from 10 Gy to 50 Gy. The alloy becomes more ductile after irradiation, exhibiting altered deformation mechanisms [15]. Furthermore, another investigation conducted by Guan et al. [16] highlighted the interaction of SAC305 with gamma rays. They discussed the underlying mechanism of beta tin (β-Sn) oxidation to tin oxide (SnO_2) due to the interaction with gamma rays. It has been revealed that oxidation initiates at the solder joint/pad interface and extends into the matrix, accompanied by the formation of micro-cracks around SnO_2/β-Sn interfaces. The findings provide valuable insights of solder alloys and gamma irradiation interaction at the atomic level offering fundamental knowledge for Pb-free solder alloy applications in electronic devices.

Although the above research focusing on microstructure and mechanical properties have been carried out and reported, thorough analysis on these characteristics induced by radiation still needs to be determined. Therefore, in this study, effect of different doses of gamma radiation towards microstructure of SAC305 solder joints were investigated. The aim of this study is to observe the evolution of the existence phases in the solder matrix, the growth of IMC layer thickness and the generation of micro-cracks due to the radiation induced defects. By achieving the objectives, this research will contribute to a better understanding on how gamma radiation interacts with the solder at atomic level and affected the solder joint at the micro level such as to their microstructure and mechanical properties.

2. Methods

In this study, 96.5Sn-3.0Ag-0.5Cu (SAC305) solder paste was used to form the solder joints. Solder paste was manually printed on the Cu substrate surface finish of the printed circuit board (PCB) by using stencil printing. Printed solder then underwent reflow soldering with the peak temperature at ~240°C. Soldered samples then cut into the small size (100 mm × 110 mm × 1 mm). The samples then go through gamma radiation exposure to determine the radiation effect on SAC305 solder joints. For gamma radiation exposure, the Gamma cell (Excel 220) gauge was used to radiate the samples by applied Cobalt-60 source. The solder was exposed to gamma radiation at different doses of 5, 50, 500, 5000 and 50000 Gy, with a specific dose rate of 0.8427 kGy/hr. After the exposure, the samples were carefully stored in a dry box to prevent any potential contamination or oxidation. The samples were then proceeded to microstructural observation and XRD analysis. Samples were subjected to metallography techniques for microstructure observation. This involved a sequence of steps including mounting, cross-sectioning, grinding, polishing, and etching. The samples were first cold mounted using epoxy and subsequently ground with silicon carbide (SiC) paper of grit sizes 400, 600, 800, and 1200. Following grinding, the samples underwent polishing using a polishing cloth and 1 μm and 0.25 μm diamond spray. Subsequently, the samples were etched using a solution of 5% hydrochloric acid and 95% methanol. Microstructural observations of soldered samples, encompassing the solder matrix and the IMC layer, were conducted utilizing an optical microscope (Eclipse M200). The average thickness of the IMC layer was measured using ImageJ software by dividing the total thickness with 100. The phase constituents of both the control sample and irradiated samples were analyzed using XRD equipment, specifically the X'Pert Pro MPD model from PANalytical, with a Cu Kα1 radiation source (λ= 1.5406 Å) and an angle 2θ in the range 20° to 80°. The collected data were then processed using X'Pert HighScore Plus to perform background subtraction, peak identification, and peak fitting. The most prominent peak corresponding to the JCPD was selected for analysis. The crystallite size (D) was estimated using the Scherrer equation:

$$D = K \times \lambda / \beta \times \cos(\theta) \tag{3}$$

where K represents the Scherrer constant (0.98), λ is XRD wavelength, β is the full width at half maximum (FWHM) and θ is the Bragg angle calculated as half of the peak position.

3. Results and discussion

Fig. 1 show the XRD spectra for unirradiated and selected gamma dose of irradiated SAC305 solder joints. Both solder joints exhibited detected peaks corresponding to β-Sn, Ag_3Sn, and Cu_6Sn_5. As a primary phase in the SAC305 solder joint, multiple peaks of β-Sn were prominently detected. No new phases were detected in the XRD spectra after exposure to gamma radiation. However, distinct changes in peak intensity were observed after radiation. The β-Sn phase in irradiated solder joints exhibits higher peak intensities compared to unirradiated solder joints. A high peak intensity may indicate a significant presence of the detected phase and offer insights into its level of crystallinity [17].

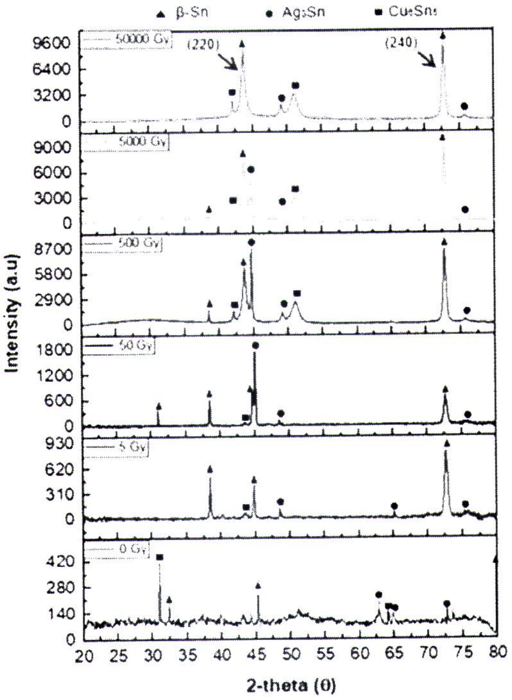

Fig. 1 XRD spectra of as-received and irradiated SAC305 solder joints.

Exposure to high dose of gamma radiation contribute to the high degree of crystallinity of β-Sn phase. Crystalline materials produce sharp and intense diffraction peaks in XRD patterns [18]. This means that the atoms in the material are arranged in a regular, repeating pattern characteristic of a crystalline structure [19]. Furthermore, additional Ag_3Sn peaks were detected as satellite peaks alongside the β-Sn (220) peaks at 0, 500, and 5000 Gy of radiation exposure. Unfortunately, at a dose of 50000 Gy, the peak was disappeared. The β-Sn (220) peaks also exhibited a noticeable shift towards lower angles. The presence of additional Ag_3Sn peaks with high intensity indicated a significant amount of Ag_3Sn in the solder matrix at 50, 500, and 5000 Gy of gamma radiation exposure. This suggests that gamma radiation accelerates the formation of the Ag_3Sn phase in the SAC305 solder matrix.

Further analysis focused on determining the crystallite size of β-Sn (220) using the Scherrer equation, which helps determine the crystallite size based on the peak of maximum intensity in each spectrum: $D = (0.9)(\lambda) / (\beta\cos\theta)$, where D, λ, β, and θ represent the crystallite size, X-ray wavelength, line broadening of the highest intensity peak in radians, and the Bragg angle, respectively. By knowing the values of the crystallite size, we were able to determine the dislocation density (δ) using the equation $\delta = 1/D^2$ [19]. Table 1 presents the tabulated data for the crystallite size and dislocation density of β-Sn (220). Initial exposure to gamma radiation increases the crystallite size of β-Sn (220) and then the crystallite size decreased at higher gamma doses, specifically at 500, 5000, and 50000 Gy. Ionization caused by gamma rays results in the creation of electron-hole pairs, which tend to accumulate around defects in the crystal structure. Additionally, this process generates heat, which can cause the overall crystal structure to reorient itself [20].

When exposed to gamma radiation, the distribution of phases exist in the SAC305 solder matrix undergoes changes. The evolution of phases due to radiation can be attributed to various factors, such as defect formation, generation of heat, and energy transfer resulting from radiation ionizing [21]. These factors influence the diffusion of Sn, Ag, and Cu atoms within the solder matrix. To investigate the impact of gamma radiation on the phases within the solder matrix, the microstructure of irradiated SAC305 solder joints was captured and analyzed using ImageJ software (Fig. 2). The dark areas in the microstructure indicate the presence of the near-eutectic phase, while the light areas represent the β-Sn phase.

Table 1 Crystallite size and dislocation density of unirradiated and irradiated SAC305 solder joint for β-Sn (220).

Gamma Radiation Dose (Gy)	**Crystallite Size (Å)**	**Dislocation Density (cm^{-2})**
0	441	5.1418×10^{-6}
5	531	3.5465×10^{-6}
50	530	3.5599×10^{-6}
500	219	2.0850×10^{-5}
5000	203	2.4266×10^{-5}
50000	240	1.7361×10^{-5}

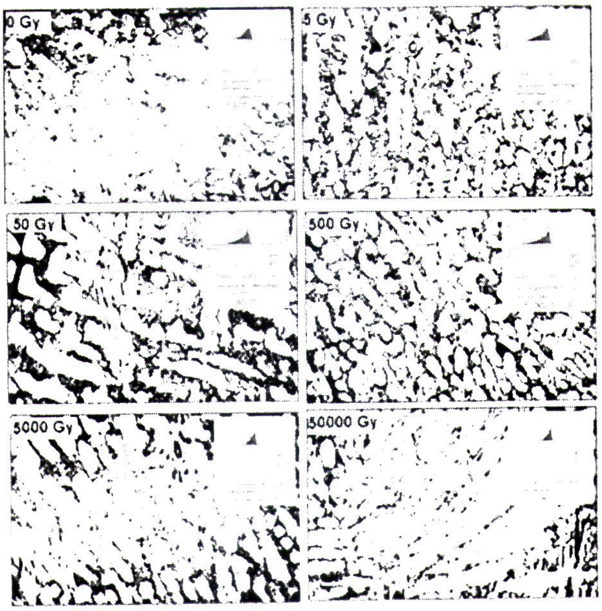

Fig. 2 Microstructure of SAC305 solder joint under different gamma radiation dose analyzed using ImageJ method.

Exposure of SAC305 solder joints to gamma-ray radiation introduces defects in their atomic structure, resulting in enhanced atomic diffusion rates attributed to the creation of vacancies and interstitials that function as diffusion pathways [22]. Consequently, this leads to increased growth and distribution of Ag_3Sn intermetallic compounds within the solder matrix. As atoms diffuse more rapidly, they tend to cluster, altering the microstructure and making it appear coarser [23-24]. However, as radiation exposure continues to increase, the accumulation of defects can reach a point where they begin to block or impede the diffusion of Ag_3Sn. These defects act as barriers that hinder the movement of atoms. Coarsening of β-Sn: As the accumulation of defects and intermetallic compound clustering progress, the β-Sn phase can start to coarsen. This means that the fine structure of the β-Sn phase becomes less refined, resulting in larger grains or structures within the solder matrix.

The interfacial IMC layer forms as a result of the chemical reaction between solder alloys and the substrate material [25]. Fig. 3 indicate the micrographs of IMC layer in SAC solder joints exposed to various gamma doses. The IMC layer in the SAC305 solder joint primarily consists of Cu_6Sn_5. The diffusion of Cu atoms into the solder bulk initiates the nucleation of Cu_6Sn_5 IMC, which subsequently grows to form the interfacial layer, as observed in the micrographs [26]. The formation of Cu_6Sn_5 intermetallic compound (IMC) is described by the following equation [7]:

$$5\ Sn + 6\ Cu \rightarrow Cu_6Sn_5 \quad (5)$$

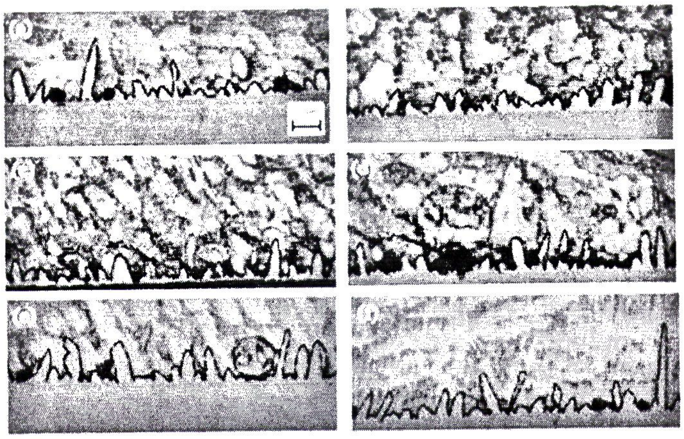

Fig. 3 Micrographs of IMC layer SAC305 solder joints under different gamma radiation dose (a) 0, (b) 5, (c) 50, (d) 500, (e) 5000 and (f) 50000 Gy.

The IMC layer exhibited a heterogeneous morphology, with scallop or needle-like structures dispersed along the solder-substrate interface, and a continuous layer formed adjacent to the solder as reported by others researcher [24-26]. Increasing gamma radiation doses had a significant impact on the thickness of the IMC layer in the solder joint. Fig. 4 illustrates that the thickness of the IMC layer increases with an increment in the gamma radiation dosage. An increase in gamma radiation dose corresponds to a longer exposure time to radiation. As the exposure time increases, it deposits more energy into the solder joint, generating heat and automatically increase the solder joint's temperature [21]. The elevated temperature accelerates the diffusion of Cu atoms into the solder bulk. The higher temperature increases the mobility of Cu atoms, facilitating their migration from the solder joint's interface into the bulk [27]. This enhanced diffusion and promoted the growth of the Cu_6Sn_5 intermetallic compound (IMC) layer. Faster growth of Cu_6Sn_5 IMC thickens the IMC layer. Therefore, the increase in radiation exposure time not only generates heat but also boosts the diffusion of Cu atoms, ultimately leading to the growth of the IMC layer in the solder joint.

Based on the graph in Fig. 4, the initial exposure to gamma radiation resulted in a slightly change of IMC layer thickness at 5 Gy dose. However, abrupt change on IMC thickness was occurred at 50 Gy and then gradually increased until 50000 Gy. When the solder joint is initially exposed to gamma radiation, the ionizing radiation creates a substantial number of defects in the material, such as vacancies, interstitials, and dislocations [28]. These defects serve as preferential nucleation sites for the growth of IMCs. As a result, there is a rapid and pronounced increase in the thickness of the IMC layer during the early stages of radiation exposure. The defects accelerate the nucleation and growth of IMC particles near the solder-substrate interface, leading to a sudden rise in the IMC layer thickness [29-30].

However, as the exposure extends, the solder system tends to reach a more stable state or an equilibrium condition. As the density of radiation-induced defects saturates, the growth rate of the IMC layer may slow down. At this point the system achieves a balance between nucleation and dissolution processes. At this stage, the gradual increase in the IMC layer thickness reflects the continuing IMC growth through conventional diffusion-limited mechanisms, similar to those observed in thermally aged systems [28,31]. Additionally, the temperature rise associated with prolonged radiation exposure influenced the growth rate of the intermetallic compound (IMC). Elevated temperatures enhanced diffusion rates and altered the kinetics of chemical reactions, contributing to the observed gradual increase in the thickness of the IMC layer.

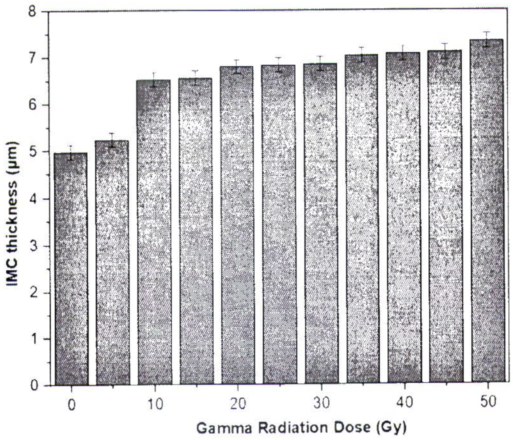

Fig. 4 IMC layer thickness of SAC305 solder joint under different dose of gamma radiation.

The ionizing radiation creates localized defects, such as vacancies, interstitials, and dislocations, within the crystal lattice of the solder material. The introduction of these defects leads to localized stress concentrations, particularly in regions near the IMC layer interface, where the material composition and crystal structure may differ [32]. IMC layer is known to possess brittle properties in comparison to the solder bulk [33]. Differences in the coefficient of thermal expansion (CTE) between the IMC layer and the solder bulk material led to the concentration of mechanical stresses at the interfaces due to temperature fluctuations induced by gamma radiation. Mechanical stress has the potential to initiate micro-cracks due to the repeated expansion and contraction of the materials [34]. Furthermore, the cracks exhibited preferential growth paths along the boundaries of the IMC layer, indicating a strong correlation between the presence of the IMC layer and crack propagation [32-33]. The combination of localized energy deposition from gamma radiation and the susceptibility of the IMC layer to radiation-induced reactions appears to play a critical role in promoting crack formation near this interface.

Correlation of microstructure evolution due to different dose of gamma radiation on micro-mechanical properties of SAC305 solder joints will be discussed. Table 2 indicates the hardness and reduced modulus of SAC305 solder joints determined by nanoindentation approach. Figure shows the bar graph of hardness versus radiation dose for SAC305 solder with as received sample and gamma irradiation samples ranging from 50 Gy to 50000 Gy. The yield strength of SAC305 solder is derived from the nanoindentation test hardness value. The obtained graph demonstrates that after being irradiated from 5 Gy to 0.5 kGy, radiosensitivity increased up to 5000 Gy before decreased. As a result of atomic displacement and transmutation products, this leads to plastic deformation, increasing hardness by more than 5000 Gy. A similar trend was observed for the reduced modulus values. According to the trend, hardness properties of SAC305 solder joint have a correlation with the distribution of near eutectic phase in the solder matrix, as revealed by the earlier analysis in microstructure. The hardness properties, indicative of a material's resistance to localized plastic deformation [35], were influenced by the formation of near eutectic phases within the solder matrix. These near eutectic phases acted as obstacles during the indentation process, leading to increased hardness values as the near eutectic percentage increased [36]. The existence of fine Ag_3Sn IMC plays a crucial role in strengthening the joint by impeding the movement of dislocations within the otherwise soft Sn matrix. The Ag_3Sn acts as a pinning effect on dislocations. However, prolonged exposure to gamma radiation leads to the coarsening of Ag_3Sn, diminishing its strengthening effect and consequently increasing dislocation mobility [36]. As a result, the hardness properties degrade with the coarsening of Ag_3Sn induced by gamma radiation.

The irradiation effects resulting from the interaction between gamma rays and materials can be categorized into three types of effects, which include Compton scattering, photoemission, and electron-positron pair production [37]. The specific type of irradiation effect is determined by both the energy of the gamma photon and the atomic number (Z) of the target material [11]. In this study, the SAC305 solder joint follows the Compton scattering effect based on the energy used for irradiation. SAC305 solder joint is irradiated by 60Co γ-ray, the energy of which is between 1.17 MeV and 1.33 MeV. Therefore, it follows the Compton scattering effect as shown in Fig. 5 [11]. The Compton scattering occurs when a powerful gamma photon hits an electron in the material, sharing energy. This makes the photon change direction and lose energy. The ejected electron, now energized, continues interacting within the material, possibly leading to radiation-induced defects.

Table 2. Hardness and reduced modulus values of unirradiated and irradiated SAC305 solder joint

Dose	Hardness (GPa)	Reduced Modulus (GPa)
Control	0.264	66.5
5 Gy	0.28	83.5
50 Gy	0.14	38.9
500 Gy	0.12	30.8
5000 Gy	0.18	40.3
50000 Gy	0.32	51.6

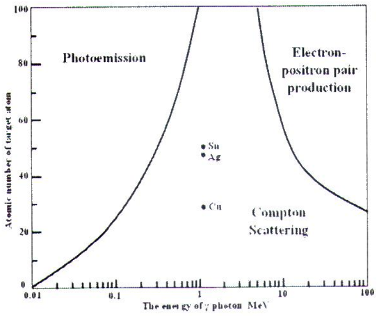

Fig. 5 Three effect domination region of γ-ray on materials [11].

4. Conclusion

In summary, different doses of gamma radiation influence the microstructure evolution of SAC305 solder joints. Significant findings are listed below:

(a) XRD spectra detected the common phase exist in the SAC305 solder joints which are β-Sn, Ag_3Sn and Cu_6Sn_5 and no new phase form after irradiation. Nevertheless, irradiated solder joints exhibited a higher peak intensity for β-Sn phase compared to unirradiated solder joint.

(b) Exposure to gamma radiation influences the near eutectic phase percentage and distribution in solder matrix. Different doses of gamma gave the different affect to microstructure evolution. At initial exposure of gamma start from 5 Gy to 500 Gy, the percentage of near eutectic area are gradually increase at the peak and then start to decrease at 5000 Gy till 50000 Gy of doses.

(c) Different doses of gamma radiation also affect the growth of IMC layer.

(d) Defects induced by gamma radiation are believed to substantially affect the microstructure evolution including the formation and distribution of β-Sn phase, near eutectic phase which reflected through the intensity peak of XRD. Microstructure evolution then significantly gave impact to the micro-mechanical properties of SAC305 solder joints.

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