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Radioactivity Control of Composite Materials Using Low Energy Photon Radiation

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1. Introduction

Composite materials (CM), often shortened to **composites,** are engineered materials made from two or more constituent materials functioning as matrix and reinforcement (Hull and Clyne 1996; Milton 2001; Strong 2007; Chung 2010). A composite material is characterized by a complex structure. Composite Materials are successfully used in various sectors of science (naval, aerospace, automotive industry and technology), replacing traditional materials such as timbers, metals and ceramic (Carlsson, Donald F. Adams et al. 2002; Strong 2007; Chung 2010). This is due to their unique attributes, such as non-existence of erosion, rust and also the fact that they demonstrate great resistibility in high temperatures. All the advantages mentioned before have made CM popular in industrial applications.

The continuously wider use of Composite Materials (CM) in various sectors of human activities imposes the growth of new methods and the construction of new appliances in order to study their physical and chemical attributes, such as density and effective atomic number, in high temperatures. The effective atomic number Z_{eff} and the density ρ are among the parameters which characterize the technological, and exploitation attributes of materials and their derivatives (Plotnikov and Pschenicniji 1973; Chudakov and Anshakov 1982; Celi 1992; Athanassiadis 1994).

Existing instruments for measuring and control of the physical and chemical properties of CM, the parameters of the technological processes don't satisfy fully the needs of research institutions and industrial enterprises. In many industrial enterprises definition of density of various materials during production is inherently confusing. The problem is particularly pronounced in developing and manufacturing of composite materials for which the density is one of the most important characteristics, directly or indirectly defines the quality composites and products from them.

The effective atomic number Z_{eff} and density ρ, parameters that play fundamental role in the behavior of materials under high temperatures are determined with the State-of-the-art techniques. These techniques determine these quantities by the average value in the total volume of the measured sample and in particular, statically after the effect of high temperature (Chudakov and Anshakov 1982).

Thus, it is expected that the creation of methods for no-contact, non-destructive control of Z_{eff} and density ρ of local parts of the material samples will allow the clarification of the

physical and mathematical models of degradation of the composite materials under conditions of high temperatures. In addition, it will assist in the improvement of the technical characteristics of parts and elements of CM, due to utilization under conditions of high temperature (up to 3000 K), and make the better possible selection of the material based on its thermal and physical properties, at the stage of product creation (Celi 1992; Athanassiadis 1994).

Radioisotope methods and appliances constitute effective means of non-destructive testing – NDT of many CM attributes. Particular characteristics, like the no-contact measurements, the high productivity, the conformity, and the relatively simple instrumentation needed, have extended the use of methods based on photon radiation transmission to the radioactive control of materials (Athanassiadis 1994). Use of low-energy sources of ionizing radiation (with an energy Eγ < 100 keV) is appropriate to improve the measurement sensitivity in controlling light environments with low thickness sensed layer (Chudakov and Anshakov 1982). Although in this case we need to take into account variations of element composition of samples.

Radiation γ-methods have comparative simplicity of implementation while providing satisfactory direction of monochromatic radiant flux. Selecting composition and energy radiation can ensure the prevalence of that kind of interaction, which contributes to maximizing information about controlled sample (Athanassiadis 1994). Depending on the effects of interaction of γ-quant with matter and scattering geometry there are resonance and non-resonance absorption methods of scattered radiation, as well as combined methods of γ-control (multi-beam methods) (Athanassiadis, Anshakov et al. 1993; Athanassiadis 2007; Athanassiadis, Chudakov et al. 2008).

2. Theory

2.1 Mass attenuation coefficient (MAC)

Mass attenuation coefficient serves as a measure of absorbing properties of the matter, characterized the interaction γ-rays with substance, and depends on its element composition and energy photons. In other worlds MAC is a measurement of how strongly a substance absorbs the radiation. Is a measure of the average number of interactions between incident photons and matter that occur in a given mass-per-unit area thickness of material encountered (Hubbell 1982).

A narrow beam of mono-energetic photons is attenuated to an intensity I from an incident intensity I_0 in passing through a layer of material with thickness x according to the exponential absorption law (Hubbell 1982) or Lambert–Beer law:

$$I_{(x)} = I_0 e^{-\mu\rho x} \tag{1}$$

where:
μ – mass attenuation coefficient (MAC),
ρ – density of the material of thickness x
Equation (l) can be rewritten as:

$$\mu = \frac{\ln(I_{(x)} / I_0)}{-\rho x} \tag{2}$$

For materials composed of multi elements the total MAC–μ is the sum of the μ_i values of each constituent element by the following mixture rule (Han, Demir et al. 2009):

$$\bar{\mu} = \sum c_i \, \bar{\mu}_i \qquad (3)$$

where:

c_i – is the concentration of individual chemical element in the sample or in other words is the proportion by weight and $\bar{\mu}_i$ is mass attenuation coefficient of the i-th element.

For materials composed of multi elements, the fraction by weight is given by (Hubbell 1982; Gigante, Pedraza et al. 1985)

$$\mu = m_A \sigma, \; m_A = \frac{\rho Z N_A}{A} \qquad (4)$$

where:

σ – the total atomic cross section, N_A- is the Avogadro's number, Z – the atomic number and A – the atomic number of element.

Considering three main mechanisms of interaction, mass attenuation coefficient μ of low energy γ-radiation in matter is the sum of (Perumallu, Rao et al. 1985; Bhandal and Singh 1993; Manohara and Hanagodimath 2007)

$$\mu = \mu_{Phot} + \mu_{Coh} + \mu_{Com} \qquad (5)$$

where:

μ_{Phot} – mass attenuation coefficient for photo effect;

μ_{Coh} , μ_{Com}– coefficients for coherent and Compton attenuations, respectively.

2.2 Effective atomic number, Z_{eff}

The atomic number, Z, is a ubiquitous parameter in atomic and nuclear physics where it occurs in almost any formula. In Composites Materials for photon interactions, the atomic number cannot be represented uniquely across the entire energy region, as in the case of elements, by a single number. For CM this number is called "effective atomic number" – Z_{eff}, and it varies with energy (Içelli, Erzeneoglu et al. 2005; Manohara, Hanagodimath et al. 2008). Z_{eff} has a physical meaning and allows many characteristics of a material to be visualized with a number (Singh, Kaur et al. 1996). This number is a very useful parameter for technology, and in many fields of scientific applications. In such applications the concept of effective atomic numbers Z_{eff} is introduced to describe the properties of these composite materials in terms of an equivalent element (Içelli, Erzeneoglu et al. 2005). The Z_{eff} is a convenient parameter for representing the attenuation of γ-rays in a complex medium and particularly in CM (Han, Demir et al. 2009). Higher values $(Z_{eff}>20)$ correspond to inorganic compounds and metals, and lower values $(Z_{eff} < 10)$ correspond to organic substances. Analysis of Z_{eff} is of a great importance for geological studies of ore and mineral composition, searching for new sources of fossil fuels, structure monitoring of composite materials, separate diagnostics of soft and bone tissues, determination of calcium content in tests for osteoporosis, etc. Study of Z_{eff} provides conclusive information about the target with which the radiation interacts (El-Kateb, Rizk et al. 2000). Z_{eff} can be determined also from the plots of atomic cross-sections versus atomic numbers of the individual elements (Mudahar, Singh et al. 1991; Parthasaradhi, Esposito et al. 1992).

The effective atomic number of a composite sample is equal (Duvauchelle, Peix et al. 1999) with the sum of equation (6). It should be mentioned, however, that the theoretical determination of Z_{eff} does not always coincide with the experimental results (Manninen, Pitkänen et al. 1984; Duvauchelle, Peix et al. 1999; Manohara, Hanagodimath et al. 2008).

$$Z_{eff} = \sum_i a_i^{at} Z_i \tag{6}$$

where:

$a_i^{at} = \dfrac{m_i / A_i}{\sum_{i=1}^{n} m_i / A_i}$ – is the gram mole fraction that is defined by the per cent of mass m_i and

the atomic mass A_i of i element.

2.3 Density measurements

γ -radiation, as mentioned, is fast, low cost, nondestructive, and easily automated. The principle used to measure density with γ -radiation is simply to pass radiation through a material attenuating the radiation, in this case gamma particles. This principle is described by the Lambert-Beer law in eq. (1) (Tiitta, Olkkonen et al. 1996)

$$I_{(x)} = I_0 e^{-\mu \rho x} \tag{1}$$

where:
$I_{(x)}$ = radiation intensity after attenuation (counts/s.)
I_o = unattenuated radiation intensity (counts/s.)
μ = mass attenuation coefficient (cm²/g)
ρ = density (g/cm³)
x = absorber thickness (cm)
With known values of μ and x, and acknowledging the existence of some amount of background radiation, I_n, Equation (1) may be rearranged as (Tiitta, Olkkonen et al. 1996; Athanassiadis 2009)

$$\rho = \frac{ln\left[\dfrac{I_0 - I_n}{I_{(x)} - I_n}\right]}{-\mu x} \tag{7}$$

The weakening of radiation depends on the thickness of a γ-rayed object and is also connected with the density of examined materials. Registering the interaction of low-energy γ-radiation with CM, we can get information about its structure and physical and chemical characteristics. Selecting composition and energy of radiation we can ensure the dominance of a particular type of interaction, to maximize the taken information of the controlled object.
Knowing the MAC of each element and element composition of analyzing object from the measured values of I and I_o, we can determine the surface density P of object (Athanassiadis 1994):

$$P = \rho l = \frac{1}{\mu_{eff}} \ln(I_0 / I) \tag{8}$$

The knowledge of natural minerals' physical parameters such as the mass attenuation coefficients M, the effective atomic number Z_{eff} is useful for understanding their physical properties.(Han, Demir et al. 2009).

It is important for densitometry of CM (Chudakov and Anshakov 1982), to define the effective parameters of γ-radiation interaction with samples of complex chemical composition. (Plotnikov and Pschenicniji 1973) proposed replacement parameters conditionally substance consisting of one element, and use expressions:

$$\frac{\rho_{eff} A_0}{A_{eff}} \psi\left(Z_{eff}, E\right) = \rho A_0 \sum_i \frac{q_i}{A_i} \psi(Z_i, E) \tag{9}$$

where q_i – content of i-th-element
$\psi\left(Z_{eff}, E\right) -$ It is accordingly:
$d\sigma^{Phot} (Z_{eff}, E)$ – for a photo effect
$d\sigma^{Coh} (Z_{eff}, E)$ – for coherent scattering and
$d\sigma^{Com} (Z_{eff}, E)$ – for Compton scattering
In the case of incoherent scattering (Plotnikov and Pschenicniji 1973):

$$Z_{eff} = \sqrt[3]{\sum_i q_i Z_i^3} \tag{10}$$

and for coherent scattering (Plotnikov and Pschenicniji 1973):

$$Z_{eff} = \sqrt{\sum_i q_i Z_i^2} \tag{11}$$

3. Non-resonance methods

3.1 Single beam γ-method

The determination of the samples density from the registration system is based on the measurement of γ-radiation beam's intensity attenuation. The beam is transmitted from a constant thickness sample. In collimated beam geometry, the intensity of γ-ray beam is given by the exponential law Eq. (1). Consequently, the density of CM is given by the relation, for samples with known values of μ and x.

$$\rho = \frac{1}{\mu x} \ln(I_0 / I) \tag{12}$$

A typical set up of the radioisotope systems to study the low energy γ-rays that transmitted through CM is given in Figure 1.

The γ-ray coming from a source S is registered by the detector D. Collimator K1 is outside of detector. Inside the detector's main body, the collimator K2, the scintillator of NaI(Tl) crystal, the photomultiplier (PMT) and the electronic block are being placed. The electronic block of radiometric data processing information, separates the informative pulses from the signal of the detector's exit. Furthermore, it stabilizes the energy scale of the signals converter and measures the pulses containing information, on a given duration time. Finally, measurements' results are presented on the PC's screen.
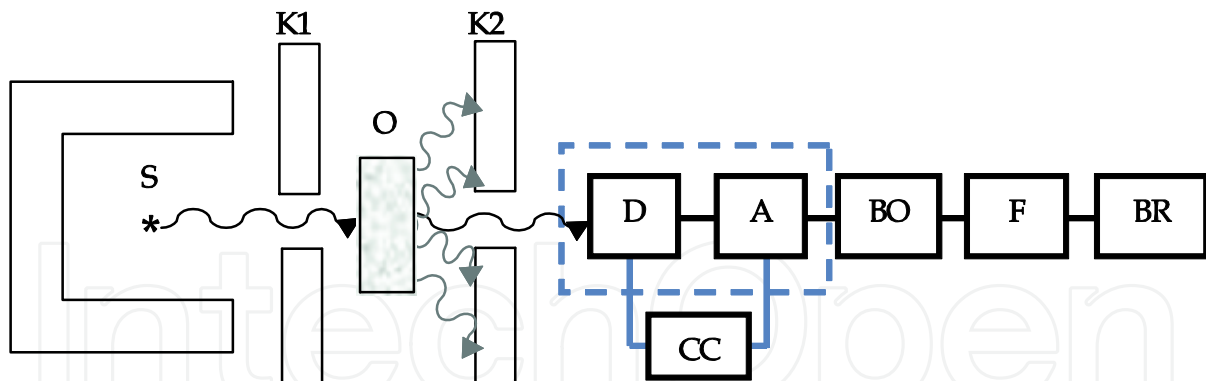
Fig. 1. A common block diagram of the single beam radioisotope measuring schemes

Ratio and system characteristics of collimators K1 and K2 or their absence, determines the required geometry measurements, which depends on the task and provides the most addictive signal on detectors output of parameters variation of the sample.

The detector is meant to realize the required measurement geometry. Additionally, it is used to achieve the registration of γ-quanta, which are transmitted from the absorber. The radiation source and the detector have been placed in specific positions in order to ensure personnel's safety. Furthermore, placing the radiation source and the detector in those positions provides the essential collimated geometry.

Density (volume density) ρ is for an object with a known thickness. In technical annexes Single beam γ-method (SBGM) received adequate development (Chudakov and Anshakov 1982; Athanassiadis 1994). SBGM in static measurements are relatively simple in his mathematical description. Single-beam γ-method is appropriate to apply when the sample thickness is x < 200 mm (Chudakov and Anshakov 1982). Optimal thickness of layer sample must, estimated on criterion of minimal statistical errors at a fixed measurement time, satisfy the condition (Chudakov and Anshakov 1982; Celi 1992):

$$l = \frac{2}{\mu P} \tag{13}$$

where:
μ – is the MAC and
$P = \rho \cdot l$ – is the surface density of a sample of thickness l and volume density ρ
(Chudakov and Anshakov 1982; Celi 1992) concludes that SBGM allows us to control a change of surface density $P=\rho \cdot l$ in the direction of the heat axis. The conclusion is based on the fact that for the control of homogeneous flat samples of CM which have been high heated from one side and as a result of that heating affected changes in all three characteristics (μ, ρ, l) that are responsible for transmission of γ-rays from an object, while the homogeneity of μ and ρ distribution characteristics is disturb in the direction of the heat. At the same time in the field of Eγ > 100 keV of CM MAC does not depend of elemental composition. We assume that decomposition of CM impact only, on reducing its density while maintaining or slight change the thickness of sample.

(Chudakov and Anshakov 1982) report that by making use of γ-densitometry methods along with the use of a single beam's radiation technology, the researcher is allowed to check the surface density's change in the address of light's combustion of Composite Materials (CM). This is due to γ-densitometry methods' great simplicity. It holds true that the CM suffers high temperature effects. The development of a system providing the

possibility to dynamically control the measurement, allows the researcher to acquire data concerning the speed of mass' loss in the unit of his heated surface. Furthermore, it offers the change to registrate the internal sample's change of density and to construct its' "behavioral" profile(Athanassiadis, Chudakov et al. 2008).

3.2 Determination of the effective atomic number Z_{eff} for composites materials
3.2.1 Theoretical aspects

The effective atomic number Z_{eff} and the density ρ are among the parameters which characterize the technological, and exploitation attributes of materials and their derivatives. Particular characteristics, like the no-contact measurements, the high productivity, the conformity, and the relatively simple instrumentation needed, have extended the use of methods based on photon radiation transmission to the radioactive control of materials (Athanassiadis 1994).

The creation of methods for no-contact, non destructive control of Z_{eff} and density ρ of localized areas of the material samples will allow the clarification of the physical and mathematical models of degradation of the composite materials under conditions of high temperatures. For the determination of Z_{eff}, it is necessary to use detectors with high energy resolution, due to the need to distinguish between spectrum lines of coherent and incoherent scattering of γ-rays (Athanassiadis 2009).

The analysis of physical and chemical characteristics of materials via the determination of change in the effective atomic number Z_{eff} can be performed by measuring the ratio of elastically N_R (Rayleigh) to the in-elastically N_C (Compton) scattered γ-rays intensities.

The total effective atomic cross-section σ_R for Rayleigh scattering approximately is:

$$\sigma_R \cong c_1 \frac{Z^n}{E^m} \tag{14}$$

where:
c_1 – constant for the particular scattering angle;
Z – atomic number;
E – energy of incident photon;
n, m – certain constant numbers.

The effective atomic number and the other parameters are independent from energy (Perumallu, Rao et al. 1985). The values of numbers n, m depend from the type of process.

Total effective atomic cross-section σ_C for Compton scattering approximately is:

$$\sigma_C \cong c_2 \frac{Z}{E^m} \tag{15}$$

Dividing the total atomic cross-section σ_R for the Rayleigh scattering (14) with the total atomic cross-section σ_C for the Compton scattering relation (15) we have the relation between the scattering cross-sections that is (Manninen, Pitkänen et al. 1984):

$$\frac{\sigma_R}{\sigma_C} = c_3 Z^{n-1} \tag{16}$$

where c_3 is a constant for the particular photon energy.

The probability of the processes of coherent and inelastic scattering and their factors are related to the atomic form factor F and the function of inelastic scattering S.

The angle dependence for differential effective cross-section $d_a\sigma_R/d\Omega$ in the unit of solid angle can be calculated multiplying the distribution function for the classic Thompson scattering in the electron and the square of atomic form factor F (X, Z), where X - the transfer momentum due to the change of trajectory of moved photon (Manninen, Pitkänen et al. 1984):

$$\frac{d_a\sigma_R}{d\Omega} = \frac{r_0^2}{2}\left(1 + cos^2\theta\right) \cdot [F(X,Z)]^2 \tag{17}$$

r_0 - the classical radius of electron.

In the coherent scattering, the transfer of momentum in the atom or in electron is performed by the change of direction of trajectory of the moving photon without change of the magnitude of the photon momentum (Karellas, Leichter et al. 1983).

The function of angle distribution for the incoherent Compton scattering is defined by the Klein-Nischina relation multiplied by the incoherent function S(X,Z) (Manninen, Pitkänen et al. 1984):

$$\frac{d_e\sigma_c}{d\Omega} = \frac{r_0^2}{2}\left[\frac{1}{1 + a(1-cos\theta)}\right]^2 \cdot \left[1 + cos^2\theta + \frac{\alpha^2(1-cos\theta)^2}{1+\alpha(1-cos\theta)}\right] \cdot S(X,Z) \tag{18}$$

Dividing (17) with (18), we obtain the relation between the cross-sections of coherent and Compton scattering as function of the photon energy, scattering angles and the scatterer's atomic number (Duvauchelle, Peix et al. 1999):

$$\frac{d_a\sigma_R/d\Omega}{d_e\sigma_C/d\Omega} = \frac{[1 + a(1-cos\theta)]^2 \cdot (1+cos^2\theta) \cdot [F(X,Z)]^2}{\left[1 + cos^2\theta + \frac{\alpha^2(1-cos\theta)^2}{1+\alpha(1-cos\theta)}\right] \cdot S(X,Z)} \tag{19}$$

This relation is important for the measurement of the atomic number with the method of Rayleigh and Compton scattering.

The exponential dependence of coherent to incoherent effective cross-sections scattering ratio from the Z^{n-1} is not obvious consequence of eq. (19), however it is expressed in functions F (Z) and S (Z) (Leichter, Karellas et al. 1985).

The values of index n depend on the value of the given scattering angle (the transfer of momentum during scattering is higher for larger values of the scattering angle). Thus, it is clear that the inner electrons, with higher binding energy, play a decisive role during the scattering process, while at lower energy levels only the outer electrons play a significant role.

More than 75% of scattering actions occur at angles smaller than the so-called "characteristic" angle:

$$\theta_0 = 2arcsin\left[0,026 \cdot Z^{1/3}\left(\frac{m_0c^2}{h\nu}\right)\right] \tag{20}$$

If an angle θ is smaller than the "characteristic" angle ($\theta<\theta_0$), then if the "characteristic" angle θ_0 approaches 0, the scattering ratio is proportional to Z^2 and does not depend on the energy E. It is important to note that the ratio σ_R/σ_C increases with increasing atomic number Z and decreasing energy E.

Taking the Thomas-Fermi approach into consideration, for the distribution of electron, the differential cross-section of the coherent scattering can be written as (Manninen, Pitkänen et al. 1984):

$$\frac{d\sigma_R}{d\Omega} = \left(\frac{d\sigma}{d\Omega}\right)_{Th} \cdot \frac{\pi Z^3}{2u^3} \tag{21}$$

where: $u = a\frac{4\pi}{\lambda}sin\frac{\theta}{2}, \alpha = \left(\frac{3}{32\pi^2}\right)^{2/3} \cdot \frac{h^2}{2mc^2}$

In this approach the differential cross-section $d\sigma_R/d\Omega$ is proportional to Z^3, however, this is only correct for scattering angles bigger than the characteristic angle (Manninen and Koikkalainen 1984; Leichter, Karellas et al. 1985).

Based on the angular frequencies of incident photons ω_1 and scattered photons ω_2^0 the Klein-Nishina formula is approximated by the relation (Manninen, Pitkänen et al. 1984; Perumallu, Rao et al. 1985):

$$\frac{d\sigma_{KN}}{d\Omega} = \left(\frac{d\sigma}{d\Omega}\right)_{Th} \cdot \left(\frac{\omega_2^0}{\omega_1}\right)^2 \tag{22}$$

and for the incoherent scattering:

$$\frac{d\sigma_C}{d\Omega} = \left(\frac{d\sigma}{d\Omega}\right)_{Th} \cdot \left(\frac{\omega_2^0}{\omega_1}\right)^2 \cdot Z \tag{23}$$

Assuming that the scattering angle is not smaller than:

$$\frac{sin\frac{\theta}{2}}{\lambda} \geq 20\,nm^{-1} or \geq 2\,A^{\circ-1} \tag{24}$$

Dividing (21) with (23):

$$\frac{d\sigma_R/d\Omega}{d\sigma_C/d\Omega} = kZ^2 \tag{25}$$

where k –numerical factor.

Equation (25) is acceptable only for specific values of energies Eγ ≤ 100 keV, scattering angles smaller than θ_0 and elements with 6 < Z < 83 (Manninen, Pitkänen et al. 1984; Athanassiadis 1994).

From this relation, one can deduce the parabolic dependence of the Rayleigh to Compton scattering ratio (R/C) from the atomic number of the scatterer (Manninen and Koikkalainen 1984; Duvauchelle, Peix et al. 1999; Manohara, Hanagodimath et al. 2008; Athanassiadis 2009).

3.2.2 Method

The atomic number of a composite sample is equal (Duvauchelle, Peix et al. 1999) with the sum of equation (6). It should be mentioned, however, that the theoretical determination of Z_{eff} does not always coincide with the experimental results (Manninen, Pitkänen et al. 1984; Duvauchelle, Peix et al. 1999).

$$Z_{eff} = \sum_i a_i^{at} Z_i \qquad (6)$$

where:

$a_i^{at} = \dfrac{m_i / A_i}{\sum\limits_{i=1}^{n} m_i / A_i}$ – is the gram mole fraction that is defined by the per cent of mass m_i and the

atomic mass A_i of i element.

The dependence of the coherent and incoherent scattering ratio N_R/N_C of the γ-radiation beams on the atomic number Z of the element, the scattering angles and the energy Eγ of radiation in the sample, can be obtained from relation (26), (Karellas, Leichter et al. 1983; Manninen, Pitkänen et al. 1984; Perumallu, Rao et al. 1985; Duvauchelle, Peix et al. 1999).

$$\frac{N_R}{N_C} \cong \frac{\sigma_R}{\sigma_C} = \frac{C_1 Z_{eff}^n / E_\gamma}{C_2 Z_{eff} / E_\gamma} \Rightarrow \frac{N_R}{N_C} = C_3 Z_{eff}^{n-1} \qquad (26)$$

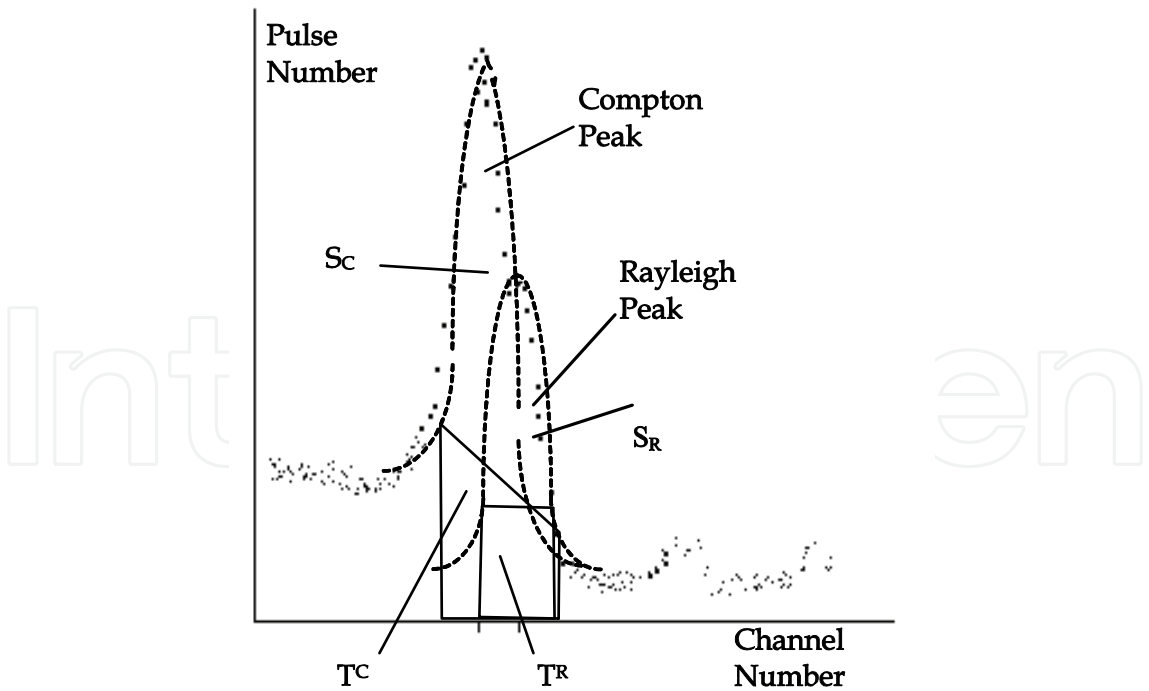where C_1, C_2 and C_3 coefficients which are not varied for the particular energy.

Fig. 2. Scattering Spectra for *Fe* obtained at scattering angle $\theta=75^0$

The theoretical determination of the values of coefficients is difficult due to the dependence of these coefficients on the model that describes the atomic shell. However, the experimental

determination of coefficients C_3 in equation (26) is straightforward. It is known (Karellas, Leichter et al. 1983; Manninen and Koikkalainen 1984; Gigante, Pedraza et al. 1985; Leichter, Karellas et al. 1985; Perumallu, Rao et al. 1985; Duvauchelle, Peix et al. 1999), that the value of exponent n for large scattering angles is: $n=3$.

The determination of Z_{eff} from the ratio N_R/N_C depends on the separation of the spectrum lines of the coherently and incoherently scattered γ-quanta. For the basic peaks of the spectrum lines of the coherent and incoherent scattering events, the variation of ratio N_R/N_C appears mainly at the distribution maximum and does not significantly influence the shape of the spectrum lines (Plotnikov and Pschenicniji 1973). However, the separation of the peaks of the spectrum lines of Rayleigh and Compton scattering is not always possible (Fig. 2). It is common to observe an overlap of the peaks. For this reason, various methods are employed for the determination of the area under the peak. This complication imposes the use of detectors with high energy resolution.

3.2.3 Other measurements

Measurements of the Rayleigh to Compton scattering ratio (Singh and et al. 2007) were used also for the evaluation of bone mineral contents and bone mineral density in a number of experimental investigations: (Puumalainen, Sikanen et al. 1979; Kerr and et al. 1980; Karellas, Leichter et al. 1983; Webster and Lillicrap 1985; Parthasaradhi, Esposito et al. 1992; Manohara and et al. 2008). These measurements have been made with radioactive sources at various scattering angles with samples of tissue or bone equivalent materials. (Speller and Horrocks 1991) and (Shakeshaft and et al. 1997) have introduced applications of this technique in the fields of medicine and biology. This technique has also been tested for some elements with atomic number satisfying, $26 \leq Z \leq 82$, by (Içelli and Erzeneoglu 2002) and (Içelli 2006).

Duvauchelle in (Duvauchelle, Peix et al. 1999; Duvauchelle, Peix et al. 2000) have concluded that the Rayleigh to Compton scattering intensity ratio depends only on the mixture under study and provides a non-destructive technique to measure the Z_{eff} of composite materials and the Z-number of unknown elements (Singh and et al. 2007). They suggested that a given Z_{eff} must define a mixture on the basis of the intensity ratio of Rayleigh to Compton scattering, as a single atom is characterized by its atomic number. This technique utilizes the strong dependence of the Rayleigh to Compton scattering intensity ratio on the effective atomic number of the scattering medium. One of the major advantages of this method is that by taking the ratio of the Rayleigh to Compton scattered photons, a number of parameters such as absolute source strength, solid angles subtended by source and the detector at the target are eliminated in the expression of the ratio technique, otherwise these parameters introduce a large amount of error in the measured results(Singh and et al. 2007).

4. Resonance methods

4.1 Theoretical aspects

In fig. 3. is shown a block diagram of Mossbauer-transmission and scattering experiments. Doppler shifted γ radiation is emitted from a source moving with a velocity v. A certain fraction of this radiation is absorbed resonantly in an absorber. The transmitted radiation is detected in a γ detector. The resonance absorption probability and thus the count rate in the detector depend on the relative velocity between the source and the absorber (Weyer 1981). By measuring the count rate at different velocities we can record a transmission spectrum.

For ideal source and absorber materials the lines in the transmission spectrum have approximately Lorentzian shapes for not too thick absorbers (Weyer 1981).
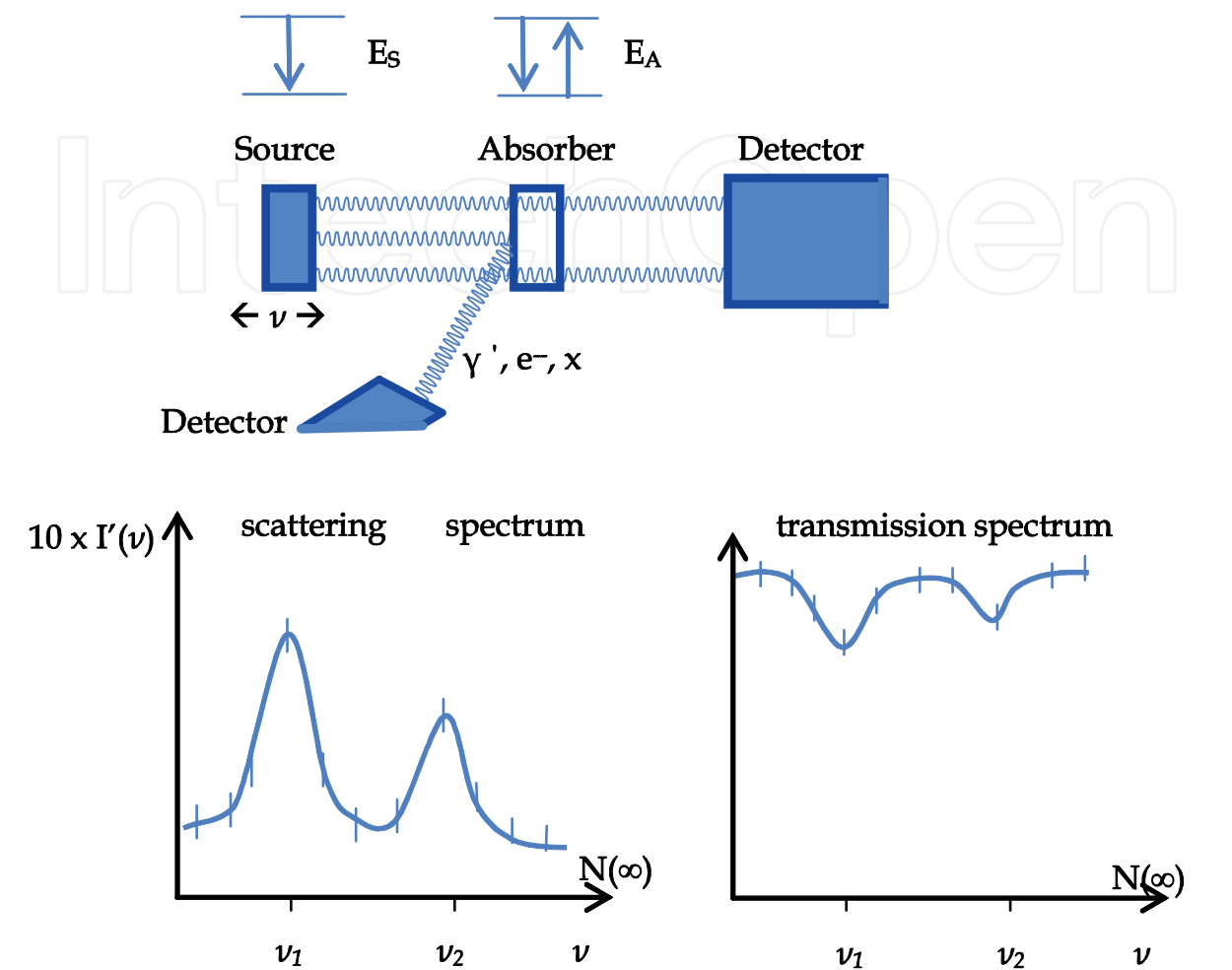

Fig. 3. Schematic illustration of a scattering and a transmission Mossbauer experiment

Mossbauer absorption spectroscopy or nuclear γ-Resonance method (NGR) in its most common form applies only to materials that contain resonance Mossbauer nucleus (such as Fe, Sn, Co) in the samples that we investigate, and this limits the usage field of the method. The problem concerning the study of physical and chemical attributes and processes in the CM and condensed matter is resolved with the method according to which the Rayleigh Scattering of Mossbauer Radiation (RSMR) is registered and in which Mossbauer radiation interacts with electrons of matter (Zolotoyabko and Iolin 1986; Mossop, Kerr et al. 1987).

4.2 Resonance detector

In 1963 Mitrofanov K.P. suggested a resonant detector which is sensitive only for recoilless gamma-quanta (Mitrofanov, Gor'kov et al. 1978). A resonant detector is a special instrument for Mossbauer spectroscopy whose principle of operation is also based on the Mossbauer Effect. The Scintillation Resonant Detector - SRD that are used in Mossbauer spectroscopy, are intended for the selective registration of resonance radiation and allows us to increase resolution and sensitivity and thus to expand the information potential of this method. The operation of resonant detectors is based on the registration of secondary radiation, primarily

secondary electrons (Belyaev, Volodin et al. 2010). In this type of detector, a resonant substance (a converter) is placed inside the working volume of the detector.

The principle of operation of SRD is illustrated in fig. 4 (Maltsev, Mehner et al. 2002). The resonant γ-quanta excite the nuclei of the grains of the resonant substance. In the case that the nucleus deexcites by emission of a conversion electron, this electron will excite along its path a number of atoms of the scintillator, which surrounds the resonant substance grain. The excited atoms of the scintillator produce photons. The photons produce photoelectrons in the photocathode of the photoelectronic multiplier tube.
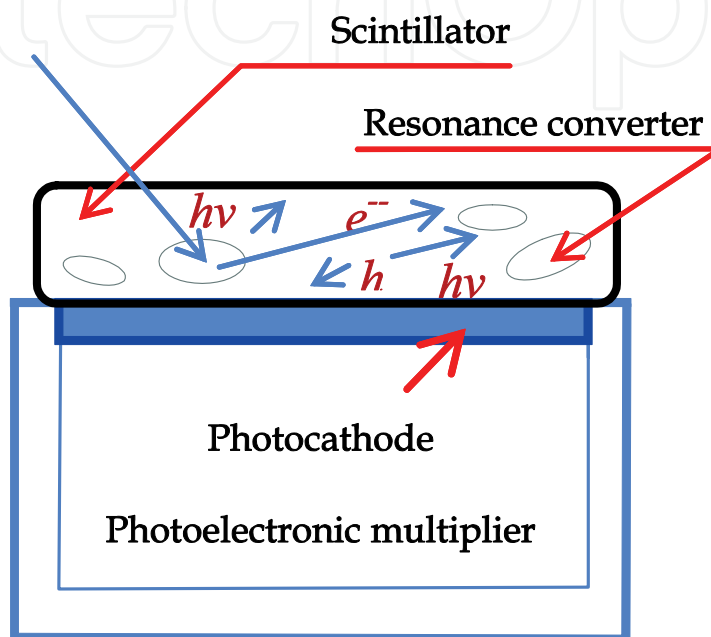

Fig. 4. Principle of operation of the Scintillation Resonance Detector - SRD

The recoilless resonant absorption of γ-rays is followed by a variety of deexcitation processes involving reemission of γ-ray, internal conversion electrons, Auger electrons and X-rays (Sawicki and Tyliszczak 1983). As a matter of fact, only the high-energy part of these deexcitation processes is utilized in Mossbauer scattering experiments, see (Bonchev, Jordanov et al. 1969; Liljequist 1981; Sawicki, Sawicka et al. 1991), whereas low-energy events represented by UV- and optical photons (Lichtenwalner, Guggenheim et al. 1976) or secondary electrons have hardly been investigated.(Sawicki and Tyliszczak 1983).

The method of resonance detection is based on the energy modulation, with moving absorber of Mossbauer radiation placed between the radioactive source (which is in resonance) and the resonance detector.

In γ- resonance spectroscopy with the use of SRD the source and the resonance detector create a resonance pair.

Basic characteristics of these pairs constitute:

- the selectivity,
- the efficiency of registration of recoilless γ-quanta,
- the permissible loading ability.

The selectivity is determined as the ratio of output of registration of γ-beams when $E = E_0$ and $E = \infty$, in case we have resonance and not, respectively. The SRD with plastic Scintillator ensure possibility of loading more than 10^6 cm^{-1}. The value of the resonant effect can be as

high as several hundred percent, when investigating the aftereffects of nuclear transformations in the emission mode (Belyaev, Volodin et al. 2010).

The substance inside the resonant detector must have (Belyaev, Volodin et al. 2010):

- a spectral absorption line width that is close to the natural width;
- a large value of the Lamb–Mossbauer factor (the *f*-factor);
- a large number of resonant atoms;
- a spectral line coinciding in the energy scale with the position of the source's spectral line. Even a small isomeric shift leads to distortion of the shape of the experimental spectrum with a loss of sensitivity and large isomeric shifts that considerably exceed the line width, leading to the disappearance of the effect.

Mossbauer spectroscopy represents one of the most informative methods for an investigation of a structure of the matter. It is well known that, an essential disadvantage of Mossbauer spectroscopy is a long time of the spectrum recording, usually of about 10 h in transmission geometry, and few tens of hours in the scattering geometry. The use of resonance detectors in Mossbauer spectroscopy allows a reliable determination of the non-resonant background level, which should be taken into account when the absolute value of the Mossbauer fraction (*f*) is needed. Simple formulae for finding the factors *f* from the areas of the experimental spectra have been derived in (Mitrofanov, Gor'kov et al. 1978). Also, the use of resonance detectors is promising way to increase a productivity of Mossbauer measurements (Mashlan, Kholmetskii et al. 2006).

4.3 Mossbauer spectrometer for the registration of rayleigh scattering of mossbauer radiation

The 75% of Rayleigh scattering actions is concentrated at angles smaller than the so-called "characteristic" angle (eq. 20) ($\theta \leq 16°$) (Manninen and Koikkalainen 1984; Duvauchelle, Peix et al. 1999). More prospects for the registration of RSMR seem to have the Scintillation Resonance Detectors – SRD (Mitrofanov, Gor'kov et al. 1978; Athanassiadis 1994; Semenov, Irkaev et al. 1995), which allow us to register relatively very small changes in energy during the Doppler modulation of energy.

Therefore, the application of the RSMR method allows us to ignore the necessity of resonance nucleus existence in the samples such as to have the ability to investigate materials, free from Mossbauer resonance nucleus, with precision that is achieved with methods of NGR ($\sim 10^{-8} - 10^{-9}$ eV) (Zolotoyabko and Iolin 1986; Athanassiadis 1994).

The methods that are based on the registration of RSMR are characterized by the relatively low intensity of the beam of secondary radiation that carries the information. Therefore, use of radiation sources of big energy or bigger duration of measurement time, is required (Semenov, Irkaev et al. 1995). These restrictions can be overridden with the creation of new types of high sensitivity detectors, among which the Combined Scintillation Resonance Detectors are distinguished, that unify the positives of various types of scintillation detectors and allow us to avoid the difficulties of multi-detector systems, difficulties that are related to their problem of inter-stabilization (Athanassiadis, Chudakov et al. 2008).

The interest for the detection techniques of secondary radiation led to new methods and detection techniques of Mossbauer radiation with the construction of new types of detectors which have the ability of registering relatively minor changes in the energy of γ-beams.

The construction of the Mossbauer spectrometer for RSMR measurement in small angles requires the study of its basic metrological characteristics such as the determination of the discrimination velocity, the peak asymmetry and the area under peak.

For SRD with radioactive isotope ^{119}Sn, dioxide of tin (SnO$_2$) is used as converter. This converter satisfies the basic requirements for converters (to have good solubility in the scintillator, in order not to decrease transparency of scintillator and so that there would be no isomeric shift in the converters spectrum relative to the Mossbauer source spectrum). The registration efficiency of Mossbauer radiation from the source ^{119m}SnO$_2$ is about 30% until 40%, while the registration efficiency of non-resonance radiation – is less than 1 %.

The spectrometer is based on the Combined Resonance Detector (Athanassiadis, Anshakov et al. 1993; Athanassiadis, Chudakov et al. 2008), which is constituted by the coaxial organic scintillation detector of 31 mm diameter and the inorganic one of CsI(Tl) of 10 mm diameter. The resonant scintillation detector consists of a thin organic film in the form of a ring, where the converter material is enriched by up to 90 % by dissolution of ^{119m}Sn Mossbauer isotope.

The spectrometer includes the movement system with constant acceleration of Mossbauer source ^{119m}Sn, the stabilization system of Combined Resonance Detector energy scale and the interface for the connection to computer PC. The system ensures the simultaneous accumulation and treatment of two spectra, which correspond in the registration of resonance radiation from the inorganic and organic scintillation detector. The signals division in the detectors' output from the two scintillators (organic and inorganic) is realized by the difference of scintillation time in the two scintillators (Athanassiadis, Anshakov et al. 1993).

For the study of CM which does not contain Mossbauer nucleus, was created an experimental system, constituted of the SRD and the automated Mossbauer spectrometer, which is given in fig. 5. The spectrometer's technical characteristics are given in table I.

The SRD measures the intensity of resonance γ - radiation which is scattered from the controlled sample. The scattered beam after its transmission from the sample was registered from resonance SRD (5). The SRD is sensitive to register only the coherent scattering of γ-radiation. The biggest of resonance absorption is observed with null velocity of γ-source movement.

The registration of Rayleigh scattering of Mossbauer Radiation (RSMR) allows a researcher to study the physical and chemical attributes along with the processes taking place inside the CM, which is free from Mossbauer resonance nucleus. This is accomplished with a remarkably high precision, by using methods of Nuclear Gamma Resonance (~ 10^{-8} - 10^{-9} eV).

The Combined Scintillation Resonance Detector CSRD is proposed for the determination of the density ρ, the effective atomic number Z_{eff} and other parameters that characterize the technological attributes of materials. These types of detectors are ideal for registering events that occur in adjacent points in space. The two scintillators are connected through the optical window with a photomultiplier.

Devices of this type are commonly known as "Phoswich detectors". The identification of the registration events in devices of this type depends on the difference between the conversion efficiency or/and the scintillations decay time constants of the scintillators that constitute the combined detector.

A phoswich detector is composed of several independent scintillation crystals coupled to a single photomultiplier tube. Generally, phoswich detectors are designed either for simultaneous detection of different radiation types or for minimizing the background radiation in a radiation field of interest. In both cases scintillation layers are considered to be a unique choice because of their relative sensitivity to the particular radiation type.
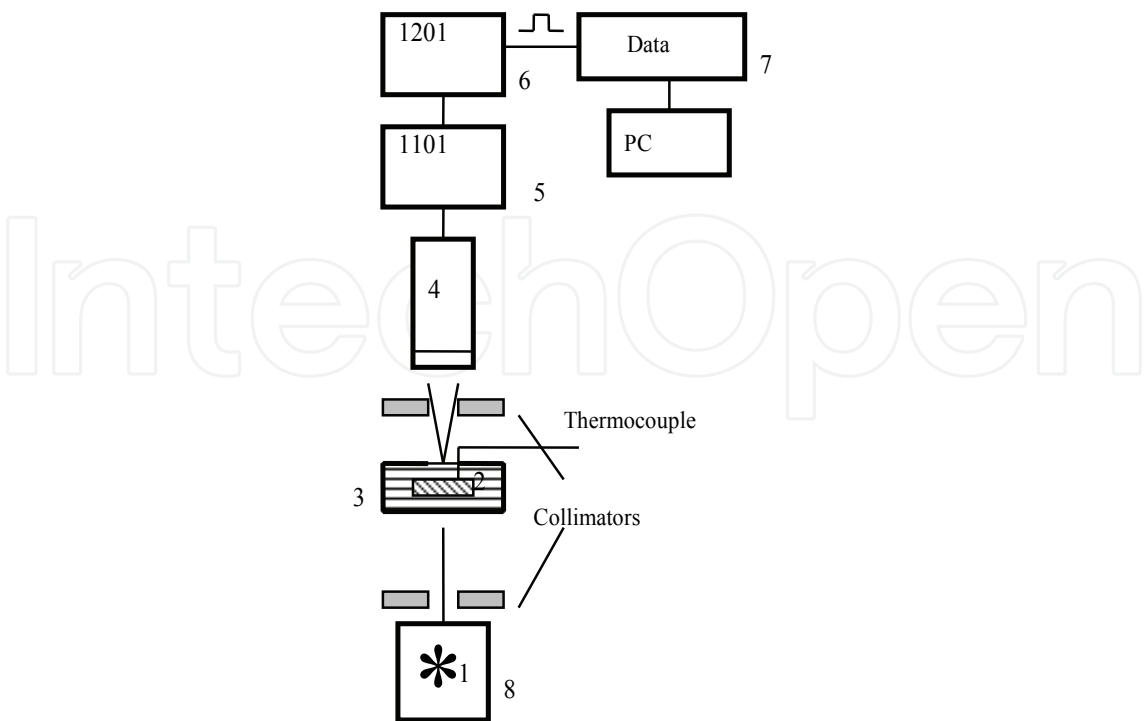
Fig. 5. Mossbauer spectrometer with Scintillation Resonant Detector

Time Stability for 24 hours of continuous work:	< 1 %
Scintillation time of NaI(Tl) scintillator:	$3 \cdot 10^{-9}$ sec
Scintillation time of Resonance scintillator:	$0.6 \cdot 10^{-6}$ sec
Amplitude ratio of Organic Resonance Scintillator to the inorganic NaI(Tl)	1 : 0.75
Discrimination Coefficient of Separation circuit:	~ 10^{-3}
Peak amplitude resolution of the inner conversion electrons:	47 %
Activity of 119mSnO2, Bq:	$8 \cdot 10^{8}$
Modulation Frequency, of γ-source motion system:	145 Hz

Table 1. Technical Characteristics of Small Angle Spectrometer of Rayleigh scattering of Mossbauer Radiation

Among other things, the scintillators are chosen to have different decay times so that the shape of the output pulse from the photomultiplier tube is dependent on the relative contribution of scintillation light from the two scintillators. The basic characteristics of the combined system are determined by the signals' separation efficiency.

5. Conclusion

By using γ- resonance and non-resonance methods for the radioactivity control of Composite Materials, we can determinate the effective atomic number Z_{eff} and density ρ. These parameters play fundamental role in the behavior of CM.

Making use of γ-densitometry methods along with the use of a single beam's radiation technology, the researcher is allowed to check the surface density's change in the address of light's combustion of Composite Materials (CM). This is due to γ-densitometry methods' great simplicity. It holds true that the CM suffers high temperature effects. The development of a system providing the possibility to dynamically control the measurement, allows the researcher to acquire data concerning the speed of mass' loss in the unit of his heated surface. Furthermore, it offers the change to registrate the internal sample's change of density and to construct its' "behavioral" profile.

These results prove the ability of the use of Mossbauer Effect, via measurements of intensity of Rayleigh scattering, for the study of dynamic physical processes in the condensed matter, which does not contain resonance nucleus, the determination of effective atomic number and the measurement of density in condensed matter(Chudakov and Anshakov 1982).

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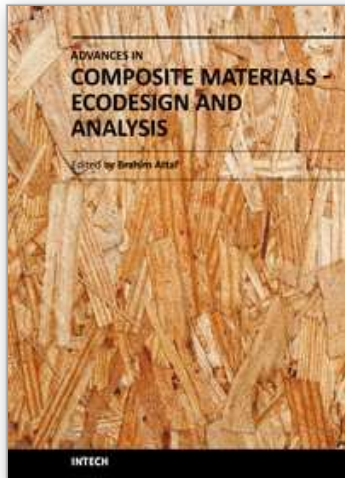
Advances in Composite Materials - Ecodesign and Analysis

Edited by Dr. Brahim Attaf

By adopting the principles of sustainable design and cleaner production, this important book opens a new challenge in the world of composite materials and explores the achieved advancements of specialists in their respective areas of research and innovation. Contributions coming from both spaces of academia and industry were so diversified that the 28 chapters composing the book have been grouped into the following main parts: sustainable materials and ecodesign aspects, composite materials and curing processes, modelling and testing, strength of adhesive joints, characterization and thermal behaviour, all of which provides an invaluable overview of this fascinating subject area. Results achieved from theoretical, numerical and experimental investigations can help designers, manufacturers and suppliers involved with high-tech composite materials to boost competitiveness and innovation productivity.

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