

# ALLOYING EFFECT ON $K$ X-RAY FLUORESCENCE CROSS-SECTION IN $\text{Cr}_x\text{Ni}_{1-x}$ AND $\text{Cr}_x\text{Al}_{1-x}$ ALLOYS

E. BÜYÜKKASAP

Atatürk University, K.K. Education Faculty, Department of Physics Education  
25240 Erzurum, Turkey

*(Received December 4, 1997; revised version February 23, 1998;  
in final form April 6, 1998)*

Alloying effect on the  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections in  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys was studied. The samples were excited by gamma rays with energy 59.5 keV from a  $^{241}\text{Am}$  radioisotope source and  $K$  X-rays emitted by the samples were counted with a  $\text{Si}(\text{Li})$  detector. We found that  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections are changed by alloying effect in  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys for different composition  $x$ . We compared our results with the theoretical values.

PACS numbers: 32.30.-r, 32.80.-t

## 1. Introduction

X-ray fluorescence cross-section is an important parameter in X-ray fluorescence analysis and various applied fields such as radiation transport in matter. In addition, comparison of measured X-ray fluorescence cross-sections with theoretical estimates provides a check on the validity of various physical parameters such as photoionisation cross-section, fluorescence yield, X-ray emission rates and jump ratio.

$K$  X-ray emission lines are caused by transition  $K_\alpha = (K \leftarrow L_{\text{II}}, L_{\text{III}})$ ,  $K_\beta = (K \leftarrow M_{\text{II}}, M_{\text{III}}, M_{\text{IV}}, M_{\text{V}}, N_{\text{II}}, N_{\text{III}} \dots)$ .  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections of various elements were measured at different energies [1-5]. Krause et al. [6] have calculated theoretically  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections. We studied chemical effect [7-9], alloying effect [10], thickness effect [11] on  $K$  emission lines intensity ratios, and chemical effect on  $L$  emission lines intensity ratios and  $L$  X-ray fluorescence cross-sections [12, 13]. Bhuinya et al. [14] and Dhal et al. [15] investigated alloying effect on  $K$  emission lines intensity ratios. Characteristic X-rays of  $3d$  elements are affected strongly by chemical structure and alloying composition.

We studied alloying effect on the  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections in  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys. We want to show that the  $K$  X-ray fluorescence cross-sections are affected by alloying composition in  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys.

## 2. Experimental

Experimental measurements were carried out on the  $K$  characteristic radiations stimulated by 59.5 keV  $\gamma$  photons of a 100 mCi  $^{241}\text{Am}$  source in pure Cr, Ni, and  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys. The samples of elements and alloys were in the form of powders. All the samples were procured from Good Fellow Company, U.K. Powdered samples were sieved by 400 mesh and prepared by supporting on the mylar film at the  $\approx 2-10 \times 10^{-3}$  g/cm<sup>2</sup> thickness. The Si(Li) detector, which has a resolution 160 eV at 5.9 keV, and ND66B pulse height analyser were used to count  $K_\alpha$  and  $K_\beta$  photons emitted from samples. The experimental setup and a typical  $K$  emission spectrum for  $\text{Cr}_{0.5}\text{Ni}_{0.5}$  are given in Fig. 1 and Fig. 2, respectively. As shown in Fig. 1, the lead shield avoids the direct exposure of the detector to radiation from the source. Iron lining on its inner side is used to absorb the  $L$  X-rays of Pb. The aluminium lining is used to collimate the  $K$  X-rays from iron shield. The  $K_\alpha$  and  $K_\beta$  peaks are resolved ones. The background is estimated by calculating the mean of ten channels before and after  $K_\alpha$  and  $K_\beta$  peaks. The peak area was found by subtraction of the background from the total peak area.

The  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections were calculated using the relation

$$\sigma_{K_i} = \frac{N_{K_i}}{I_0 G \varepsilon_i \beta_i m_j}, \quad (1)$$

where  $N_{K_i}$  is the intensity observed for the  $K_i$  emission line of the element,  $I_0$  is the intensity of exciting radiation,  $G$  is the geometry factor,  $m_j$  is the mass of the element in sample (g/cm<sup>2</sup>),  $\beta_i$  is the target self-absorption correction factor for both the incident and emitted radiation and  $\varepsilon_i$  is the detection efficiency of the detector at the energy of  $K_i$  lines.

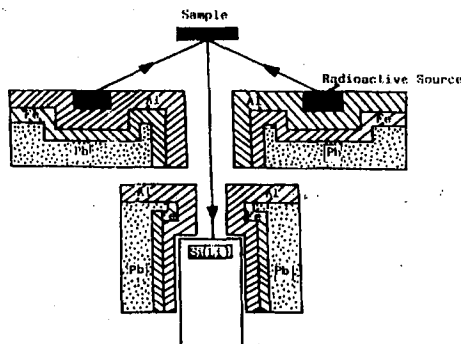


Fig. 1. Experimental geometry.

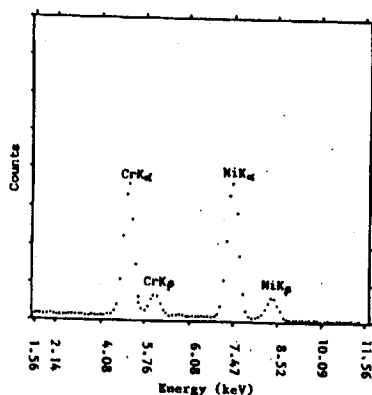


Fig. 2. A typical spectrum for  $\text{Cr}_{0.5}\text{Ni}_{0.5}$ .

$I_0G\varepsilon$  values in the present experimental setup were determined in a separate experiment. Targets of pure elements, having areas of cross-section similar to those used in the main experiment, with atomic number  $22 \leq Z \leq 40$ , emitting fluorescent radiation in the energy range 4.5–18 keV were irradiated in the same geometry and fluorescent radiation were counted.  $I_0G\varepsilon$  values for the present setup were determined by the following relationship:

$$I_0G\varepsilon = \frac{N_{K_i}}{\sigma_{K_i}\beta_i m_j}, \quad (2)$$

where  $N_{K_i}$  is the number of counts under the  $K_\beta$  or  $K_\alpha$  peaks;  $\sigma_{K_i}$  is the  $\sigma_{K_\beta}$  or  $\sigma_{K_\alpha}$  fluorescence cross-section.

The self-absorption correction was calculated by using the following expression obtained by assuming that the incidence angle of the fluorescent X-rays subtended the detector to be approximately  $90^\circ$  [16]:

$$\beta(\text{emt}) = \frac{1 - \exp\{(-1)[\mu(\text{inc})/\cos\phi + \mu(\text{emt})]t\}}{[\mu(\text{inc})/\cos\phi + \mu(\text{emt})]t}, \quad (3)$$

where  $\mu(\text{inc})$  [ $\text{cm}^2$ ] and  $\mu(\text{emt})$  [ $\text{cm}^2$ ] are the mass absorption coefficients [17] at the incident photon energy and fluorescent energy of the sample, respectively;  $t$  (in mass thickness unit  $\text{g}/\text{cm}^2$ ) is the measured thickness of the sample. Angle  $\phi$  was calculated by using the following expression [18]:

$$\cos\phi = \frac{\nu}{[\nu^2 + 0.25(R_0 + R_1)^2]^{1/2}}, \quad (4)$$

where  $\nu$  is the distance from the source to sample,  $R_0$  and  $R_1$  are internal and external diameters of radioisotope source, respectively. Angle  $\phi$  was found equal to  $48^\circ$  (Fig. 1). In addition to this, we also checked the angle, from the shift in energy of the incoherent scattered peak from the coherent peak and using Compton scattering formula.

In the case of  $\text{Cr}_x\text{Ni}_{1-x}$  alloy, there is an effect of enhancement by about 3–6% of the Cr K emission lines intensity by the K emission from the Ni. This effect was taken into account [19–21].

## 3. Results and discussion

In Table we compared the  $K_\alpha$  and  $K_\beta$  X-ray fluorescence cross-sections in different  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys. Possible sources of error include: (a)  $I_0G_e$  determination (1%), (b) background determination (0.5%), (c) self-absorption (0.5%), (d) counting statistics (0.5%). Adding these sources of error, the total experimental relative error is 2.5%.

TABLE

$K$  X-ray fluorescence cross-sections of Cr and Ni in  $\text{Cr}_x\text{Ni}_{1-x}$  and  $\text{Cr}_x\text{Al}_{1-x}$  alloys and pure metals (barns/atom).

$\text{Cr}_x\text{Ni}_{1-x}$					$\text{Cr}_x\text{Al}_{1-x}$			Theoretical values [6]			
$x$	Cr		Ni		$x$	Cr		Cr		Ni	
	$\sigma_{K_\alpha}$	$\sigma_{K_\beta}$	$\sigma_{K_\alpha}$	$\sigma_{K_\beta}$		$\sigma_{K_\alpha}$	$\sigma_{K_\beta}$	$\sigma_{K_\alpha}$	$\sigma_{K_\beta}$	$\sigma_{K_\alpha}$	$\sigma_{K_\beta}$
0.0	—	—	33.55	4.28	0.0	—	—	14.5	1.64	40.3	4.8
			$\pm 0.83$	$\pm 0.11$							
0.5	19.53	2.38	27.01	3.66	0.6	10.29	1.42				
	$\pm 0.48$	$\pm 0.06$	$\pm 0.67$	$\pm 0.09$		$\pm 0.26$	$\pm 0.04$				
0.8	15.98	2.11	29.89	5.15	0.8	15.32	1.70				
	$\pm 0.40$	$\pm 0.05$	$\pm 0.74$	$\pm 0.12$		$\pm 0.38$	$\pm 0.04$				
1.0	15.62	1.95	—	—	1.0	15.62	1.95				
	$\pm 0.39$	$\pm 0.04$				$\pm 0.39$	$\pm 0.05$				

In the  $\text{Cr}_x\text{Ni}_{1-x}$  alloys, we observed an increase in the values of  $\sigma_{K_\beta}$  of chromium with decreasing chromium concentration. There is an increase in the values of  $\sigma_{K_\beta}/\sigma_{K_\alpha}$  ratio of the nickel with decreasing nickel concentration ( $\sigma_{K_\beta}/\sigma_{K_\alpha}$  values of nickel are equal to 0.128, 0.135, 0.172 for nickel concentrations 1, 0.5, 0.2, respectively).  $\sigma_{K_\beta}$  cross-section of nickel and  $\sigma_{K_\beta}/\sigma_{K_\alpha}$  ratios of chromium have different values for different alloys compositions. The reason of this effect may be that the electronegativity of nickel is higher than that of chromium and electron transfer from the outermost orbitals of chromium to the outermost orbitals of nickel occurs. In addition to this, there are five  $3d$  electrons in chromium, whereas there are more than nine  $3d$  electrons in nickel in solid form.

In the  $\text{Cr}_x\text{Al}_{1-x}$  alloys, there is an increase in  $\sigma_{K_\beta}$  cross-sections values of chromium with increasing chromium concentration. The reason of this effect may be that the electronegativity of chromium is higher than that of aluminium and electron transfer from aluminium to the outermost orbitals of chromium occurs.  $\sigma_{K_\beta}/\sigma_{K_\alpha}$  ratios of chromium have different values for different alloys compositions.

The transitions concerned with  $K_\beta$  lines were affected directly by changes in the distribution of outermost valence electrons. Changes in the distribution of outermost valence electrons cause a shift of energy levels in the atom with respect to molecular orbital theory. The transitions concerned with  $K$  lines were affected by this event. There are clear differences between  $K$  X-ray fluorescence cross-section values of alloys and pure chromium and nickel. The values of  $\sigma_{K_\beta}$  and

$\sigma_{K\alpha}$  cross-section for pure chromium and nickel are in agreement with theoretical values [6].

In conclusion, we say that alloying effect in Cr, Ni, and Cr, Al alloys could have altered the X-ray fluorescence cross-sections of these elements from the free-atom values. To reach a more definitive conclusion about alloying effect on X-ray fluorescence cross-section, we plan to extend these measurements for alloys of various 3d metals and various concentrations.

### References

- [1] M.L. Garg, D. Mehta, S. Kumar, P.C. Mangal, P.N. Trehan, *X-Ray Spectrom.* **14**, 165 (1985).
- [2] A. Rani, R.K. Koshal, S.N. Chatuvedi, N. Nath, *X-ray Spectrom.* **17**, 53 (1988).
- [3] N.S. Saleh, A.J. Abu El-Haija, *J. Phys. B* **21**, 3077 (1988).
- [4] S. Singh, R. Rani, D. Mehta, N. Singh, P.C. Mangal, P.N. Trehan, *X-Ray Spectrom.* **19**, 155 (1990).
- [5] S. Çolak, E. Büyükkasap, Ö. Şimşek, H. Erdoğan, *Doğa Tr. J. Phys.* **17**, 806 (1993).
- [6] M.O. Krause, C.W. Nestor, Jr, C.J. Sparks, Jr, E. Ricci, Oak Ridge National Laboratory Report, Tennessee, ORNL 5399 (1978).
- [7] A. Küçükönder, Y. Şahin, E. Büyükkasap, *J. Radioanal. Nucl. Chem.* **170**, 125 (1993).
- [8] A. Küçükönder, Y. Şahin, E. Büyükkasap, A. Kopya, *J. Phys. B* **26**, 101 (1993).
- [9] A. Küçükönder, Y. Şahin, E. Büyükkasap, *Nuovo Cimento D* **15**, 1295 (1993).
- [10] Ö. Sögüt, E. Büyükkasap, A. Küçükönder, M. Ertuğrul, Ö. Şimşek, *Appl. Spect. Rev.* **30**, 175 (1995).
- [11] E. Büyükkasap, *Appl. Spect. Rev.* **32**, 151 (1997).
- [12] Ö. Sögüt, E. Büyükkasap, A. Küçükönder, M. Ertuğrul, *Appl. Spect. Rev.* **32**, 167 (1997).
- [13] E. Büyükkasap, *Spectrochim. Acta B* **52**, 1167 (1997).
- [14] C.R. Bhunya, H.C. Padhi, *Phys. Rev. A* **47**, 4895 (1993).
- [15] B.B. Dhal, H.C. Padhi, *Phys. Rev. A* **50**, 1096 (1994).
- [16] L.F.S. Coelho, M.B. Gaspra, J. Eichler, *Phys. Rev. A* **40**, 4093 (1989).
- [17] J.H. Hubbell, S.M. Seltzer, U.S. Department of Commerce Technology Administration, National Institute of Standards and Physics Laboratory, Gaithersburg, NISTIR 5692 (1995).
- [18] A. Zararsiz, E. Aygün, *J. Radioanal. Nucl. Chem., Articles* **129**, 4093 (1989).
- [19] E.B. Bertin, *Principles and Practice of X-Ray Spectrometric Analysis*, Plenum Press, New York 1975.
- [20] R. Tertian, F. Claisse, *Principles of Quantitative X-Ray Fluorescence Analysis*, Heyden, London 1982.
- [21] R. Jenkins, *An Introduction to X-Ray Spectrometry*, Wiley, New York 1986.