

CONTAMINATION OF THE CHARACTERISTIC X-RADIATION OF
COPPER BY Fe, Cr AND Ni RADIATION

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During the course of x -ray investigations on tungsten carbide and on mica crystals, reflections were noted on Weissenberg photographs which suggested the presence of superlattices. Further study led the authors independently to attribute these reflections to a contamination of the characteristic copper radiation with wavelengths characteristic of iron, chromium, and nickel. Although investigators are quite aware of poten-

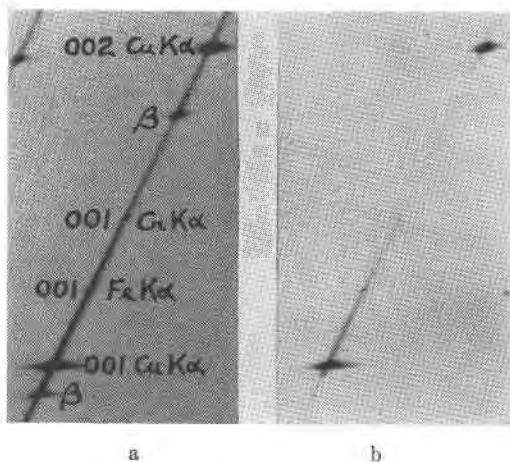


FIG. 1. Portion of Weissenberg photograph showing $(00l)$ of WC crystal taken with (a) unfiltered Cu radiation, and (b) Cu radiation filtered through nickel foil; 35 KVP, 15 MA, 15° -oscillation, 12 hrs. (overexposed for reproduction, $2\times$.)

tial contamination of the target by tungsten, we find only a brief statement in the crystallographic literature of difficulties arising from “. . . iron and other metals . . .”¹ In addition to the two x -ray tubes used by the authors, several other diffraction tubes of the same manufacturer are known to give this effect.

Figure 1 shows a portion of the $(00l)$ central lattice line of a tungsten carbide (WC) crystal taken with (a) unfiltered copper radiation, and (b)

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¹ Peiser, H. S., Rooksby, H. P., and Wilson, A. J. C. (1955), *X-ray diffraction by polycrystalline materials*, London, Institute of Physics, p. 62.

copper radiation filtered through nickel foil. The (001) reflections due to $\text{FeK}\alpha$ and $\text{CrK}\alpha$ can be clearly seen in (a); that due to $\text{NiK}\alpha$ was too weak to reproduce. The nickel filter virtually eliminated the chromium radiation but permitted a considerable portion of the iron radiation to diffract. Misinterpretation of such a pattern as evidence of a superlattice is possible, since the contaminating wavelengths cause reflections to occur for one-quarter and one-half the true Weissenberg spacing. The absence of any such reflections between the undiffracted beam and the spot (001) serves to invalidate a superlattice interpretation.

The contaminating wavelengths are primary in origin; that is, they originate at the target of the x -ray tube and cannot be attributed to fluorescence. An agent of the manufacturer has stated that "... Cr, Fe and Ni ... emanate from the stainless steel structure comprising the cathode supports and focusing cup." The latter was determined by chemical analysis to consist of approximately 75% iron and 25% chromium. The source of nickel is probably the disc-like shield surrounding the filament leads and located immediately beneath the cathode supporting block. A cursory chemical analysis showed this shield to consist of better than 95% nickel. Two of the x -ray tubes mentioned, after only 24 hours of use, gave photographs on which reflections due to iron and chromium were distinctly evident. One of these tubes was checked after 360 hours of operation, and the photograph obtained showed that the reflections due to iron and chromium had increased noticeably in intensity relative to those of copper.

The problem of preventing the migration of elements within a sealed x -ray tube operated under conditions of high vacuum, temperature, and applied voltage is at best a difficult one. It is by no means the purpose of this note to point out a "defect" in tube construction but to stress that the supposition of a superlattice must be considered in light of possible contamination of the x -ray target. Such diffraction by spurious radiation is not necessarily eliminated by filtering in all cases, and may appear on powder patterns as well as photographs made by single-crystal methods.

Although our tubes were obtained from the same source, some workers in x -ray diffraction apparently have been aware of what appears to be similar contamination for some time in tubes of another prominent manufacturer.