

SMALL-ANGLE X-RAY SCATTERING BY METALS

A conference report by W. H. Robinson

AN important event on the science calendar of Kansas City, Missouri, occurred last fall when the International Conference on Small-Angle X-Ray Scattering by Metals was held at the Midwest Research Institute on September 23–25, 1958. It was the first major scientific conference to be held in Kansas City, and it resulted, indirectly, from the intense interest of Kansas City's civic leaders in having their city recognized as a scientific center as well as for its steaks.

For the past three or four years, it has been quite evident to the person who has followed the reports on the small-angle scattering of x rays by metals subjected to various types of cold working that there has been little agreement on the source of the scattering. The experimental results have been variously interpreted in terms of scattering by submicroscopic clusters of vacancies, dislocations, or a double Bragg process, with a bit of surface scattering thrown in for good measure. The widespread interest in developing a technique for determining the density of dislocations in the volume of a plastically deformed

crystal attracted several investigators to the study of the small-angle scattering of x rays by cold-worked metals. Other researchers have used the small-angle scattering technique in seeking to determine the possible role of submicroscopic cavities in the fatigue failure of metals.

The need for clarification of the mechanism of this small-angle scattering was recognized by a group of scientists studying fatigue failure in metals at Midwest Research Institute in Kansas City, Missouri, and early in 1958 they proposed a meeting devoted to this single topic in order to consider the results of recent work on scattering and its interpretation. A Conference Committee, headed by J. C. Grosskreutz and including C. S. Barrett, W. W. Beeman, and P. A. Beck, organized the international conference around ten invited papers, with registration limited to about fifty in order to permit full discussion of the papers and the interchange of ideas. Of the ten invited speakers, six were from Europe and four were from the United States. The conference was sponsored by the National Science Foundation, Office of Ordnance Research, Office of Naval Research, and Wright Air Development Center, United States Air Force.

The host institution, Midwest Research Institute, is

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Participants in the International Conference on Small-Angle X-Ray Scattering by Metals.

Front Row: Paul W. Schmidt, U. of Missouri; James F. McGee, St. Louis U.; Maurice B. Webb, GE Research Laboratory; H. H. Atkinson, Cavendish Laboratory, Cambridge, Eng.; Peter A. Franken, Bolt Beranek & Newman, Inc.; Watt W. Webb, Union Carbide Corp.; Helmut Weik, Wright Air Development Center; Ralph T. Johnson, Jr., Kansas State Col.; and T. Bruce Daniel, U. of Kansas.

Second Row: Robert T. Howard, U. of Kansas City; R. E. Smallman, AERE, Harwell, Eng.; Charles S. Barrett, U. of Chicago; Paul A. Beck, U. of Illinois; Robert M. Bragg, Armour Research Found.; Sheldon L. Levy, Midwest Research Inst.; Andre Guinier, U. of Paris, Fr.; Ernst Bauer, US Naval Ordnance Test Station; John M. Sivertsen, U. of Minnesota; Robert Friauf, U. of Kansas; Volkmar Gerold, Max-Planck Inst. für Metallforschung, Stuttgart, Ger.; and Kenneth L. Yudowitch, Johns Hopkins U.

Third Row: Alfred Seeger, Max-Planck-Inst. für Metallforschung; A. Franks, National Physical Laboratory, Teddington, Eng.; J. S. Koehler, U. of Illinois; Paul J. Bryant, MRI; Howard E. Sorrows, Office of Naval Research; William M. Mueller, Denver Research Inst.; G. W. Brady, Bell Telephone Laboratories; Walter T. Ogier, U. of California; Richard E. Marburger, General Motors Corp.; Walter J. Trapp, WADC; James P. Dietz, General Electric Co.; W. W. Beeman, U. of Wisconsin; and Melvin T. Lavik, MRI.

Back Row: William G. Brammer, Atomics International; Harold Bale, U. of Missouri; W. H. Robinson, Carnegie Inst. of Technology; H. Brumberger, Syracuse U.; Robert Wild, U. of California; Gordon Gross, MRI; J. Charles Grosskreutz, MRI; Ralph O. Simmons, U. of Illinois; A. W. Kenneth Metzner, Cornell U.; R. Dean Dragsdorf, Kansas State Col.; B. S. Borie, Oak Ridge National Laboratory; Fred R. Rollins, MRI; and Glen E. Conklin, U. of Kansas.

a relatively young organization, having been established in 1945. It is an independent, nonprofit organization conducting research on a contract basis for industrial, commercial, agricultural, and national defense interests. Arrangements were made by MRI for the conferees to be lodged on an entire floor of an air-conditioned dormitory on the University of Kansas City campus, within easy walking distance of the Institute, thus facilitating the informal discussions and visiting among the registrants which naturally occurs at any conference. The Conference Committee and MRI are to be heartily congratulated for the completeness and attention to detail with which the con-

ference was planned and managed. The hospitality of MRI in arranging for many interesting diversions between sessions, such as tours of the laboratory and the nearby Linda Hall Library, a fine banquet, and even tickets to a baseball game between the Kansas City Athletics and the Cleveland Indians (especially interesting for some of the European visitors) attests to the fact that Midwest Research Institute and Kansas City have much to offer as a site for future scientific meetings. Particularly unique in the setting of the conference was the very modern Linda Hall Library where the first day of sessions was held. The walnut-paneled, air-conditioned structure houses an



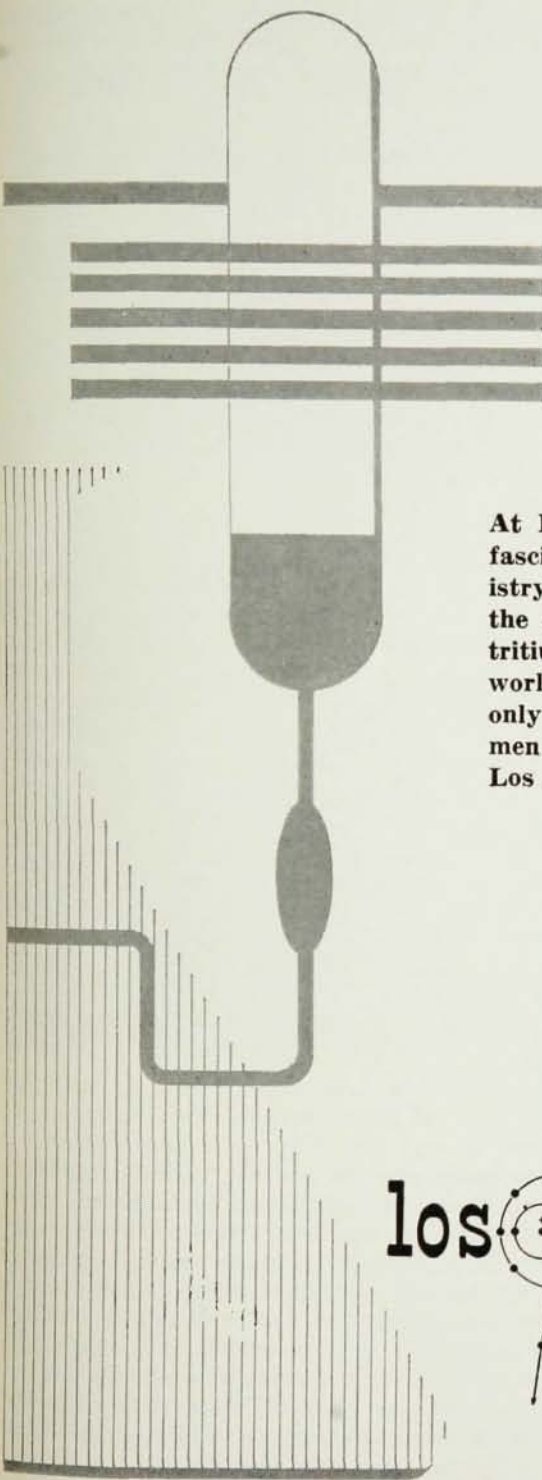
Six of the speakers were from Europe. Shown left to right are: A. Guinier from France; A. Franks, H. H. Atkinson, and R. E. Smallman from England; and Volkmar Gerold and Alfred Seeger from Germany. At far right, J. Charles Grosskreutz, Chairman of the Conference Committee, MRI. Photo by David Cauthen, The Kansas City Star

impressive library comprising 50 000 up-to-date books of science and technology (published mostly within the last ten or fifteen years), about 150 000 volumes of scientific serial publications (some of them extend back 200 to 300 years!), and a current subscription list of nearly 7000 periodicals of science and technology. This fine collection of titles, together with complete microfilming and photocopying facilities, make this privately endowed, free library a valuable asset to Kansas City's effort to become an important center of science and technology.

THE first session of the conference began with a paper by W. W. Beeman on "Experiments on the Mechanism of Small-Angle Scattering" in which he described the interpretation of a series of experiments begun in 1955 on the small-angle scattering of x rays by cold-worked metals in terms of double Bragg scattering. Beeman and his group were the first to suggest this mechanism as the source of the small-angle scattering from cold-worked metals, and it seemed quite appropriate that he provided the "key-note" of the conference. The results of these experiments on the temperature dependence of the intensity of the small-angle scattering, the immediate appearance of the scattering upon cold working at liquid nitrogen temperatures, and the polarization of the scattered x rays left little room for doubt that double Bragg scattering could account for the observed small-angle scattering. The second paper on "The Role of the Double Bragg Diffraction on Small-Angle Scattering" was presented by A. Guinier, who discovered the effect of small-angle x-ray scattering by cold-worked metals nearly twenty years ago. Since double Bragg

scattering cannot be avoided in crystalline materials, Guinier investigated the intensity variation of doubly diffracted x rays in nearly perfect crystals in order to determine whether additional useful information concerning the nature of the deformation of the crystal could be obtained. The rather distressing result was that the information obtained from the doubly diffracted x rays could be gotten better from a singly diffracted beam, and that the double Bragg scattering appeared to be mostly a nuisance effect masking real small-angle scattering caused by heterogeneities in the electron density of the specimen. The first session was brought to a close with a paper by A. Franks on the "Study of the Low-Angle X-Ray Scattering from Single Crystals in a Polycrystalline Foil". The results of this work in which large-grained polycrystalline foils were subjected to fatigue damage clearly showed that the observed small-angle scattering was due to double Bragg scattering and not to submicroscopic cavities which might be formed during fatiguing.

After lunch there was a conducted tour through the Linda Hall Library, following which the second session of the conference was opened with a paper presented by W. H. Robinson on "The Small-Angle Scattering of X Rays by Plastically Deformed Single Crystals" by W. H. Robinson and R. Smoluchowski. In this work it was found that appreciable small-angle scattering occurred only for the deformed single crystals which had twins in the irradiated volume of the sample and this effect was interpreted in terms of double Bragg scattering. It was also observed that surface scattering was an order of magnitude greater than the scattering from 10^{12} edge dislocations per cm^2 as calculated from theory. In a paper by V. Gerold

A large, stylized illustration of a chemical apparatus on the left side of the page. It features a vertical tube with a bulb in the middle, connected to a horizontal tube with several parallel lines extending from it. Below the bulb is a U-tube. To the left of the U-tube is a series of vertical lines of varying heights, resembling a chromatogram or a set of test tubes.

chemistry

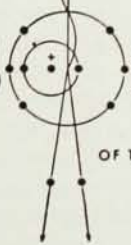
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and H. Fricke on "Experiments on Double Bragg Reflections in Deformed and Undeformed Metal Foils", V. Gerold described a very extensive series of experiments designed to study double Bragg reflections directly. With an impressive series of photographs, which registered some of the large-angle reflections, as well as the small-angle region, he was able to show directly that strong scattering occurred in the small-angle region due to $(hkl) - (\bar{h}kl)$ reflections in deformed mono- and polycrystalline foils, and that the intensity from these double reflections was considerably weaker for undeformed crystals. Double Bragg reflections of the type $(h_1k_1l_1) - (h_2k_2l_2)$, which do not appear in the small-angle region, were also obtained. The first day of sessions was concluded with a paper by J. C. Grosskreutz and F. R. Rollins on "Small-Angle Scattering from Aluminum and Copper under Cyclic Stress" given by J. C. Grosskreutz. The conclusions drawn from this work were that the small-angle scattering from fatigued metals could be accounted for by the double Bragg reflection mechanism, that no evidence for submicroscopic cavities which might lead to fatigue failure could be found, and that small-angle scattering from dislocations is an order of magnitude less than the observed scattering.

Entertainment for the evening consisted of cocktails and a banquet dinner at the "Carriage Club" in the fashionable "country club section" of Kansas City. An after-dinner talk on the "History of Communications Among Scientists, Including Some Modern Problems" was given by Joseph Shipman, Librarian of the Linda Hall Library, in which he underscored the immensity of the problem of a scientist keeping abreast of the literature in even a restricted field of science.

THE second day's sessions began with a summary of the theory of the small-angle scattering by dislocations,¹ which is probably the best theoretical treatment of the phenomenon available, and a description of some experimental work on "Low-Angle X-Ray and Neutron Scattering from Deformed Metals" by H. H. Atkinson. The remarkable agreement between the double Bragg scattering pattern that was predicted by plotting a stereogram and the picture that was obtained from polycrystalline foils was convincing evidence of the major role which this effect plays in the small-angle scattering from cold-worked metals. An ingenious neutron scattering experiment was described which showed conclusively that dislocation scattering contributed a negligible amount to the small-angle scattering from cold-worked metals which has been observed by many investigators.

It can be seen that the conference was devoted strictly to the small-angle scattering from cold-worked metals up to this point. A bit of variety in the program was provided by a paper on "Low-Angle Scattering from Quenched and Irradiated Metals" by R. E. Smallman in which "true" small-angle scattering,

not attributable to double Bragg scattering, was observed from metal foils which had been quenched and aged. Presumably the scattering was due to some sort of aggregates of vacancies. Electron microscopy suggested that these aggregates were dislocation loops in aluminum and copper (with these loops possibly causing sufficient distortion of the lattice so that some double Bragg scattering might have occurred), while for silver and gold, which have lower stacking fault energies, the vacancies seemed to be collected into closed tetrahedra of stacking faults. At an informal gathering the morning after the conference Smallman also presented some interesting results on small-angle scattering from lithium fluoride, copper, and aluminum irradiated by neutron or alpha particles. J. S. Koehler added to the variety by discussing some results of small-angle scattering experiments on deuterium irradiated germanium. The terms "cold-worked metals" and "double Bragg scattering" were not even mentioned! The observed scattering was interpreted in terms of a cloud of vacancies and a cloud of vacancies separated by a distance " a " which constituted a sort of dipole scattering.

THE last session of the conference began with a discussion by A. Seeger on the "Small-Angle Scattering from Dislocations: Present State of the Theory and a Comparison with Experiments", in which he itemized the optimum experimental conditions for observing the small-angle scattering from dislocations. In a review of the theory, Seeger showed that scattering from a normal distribution of dislocations would not be observed in the usual small-angle scattering experiments, but under certain conditions it might be possible to observe scattering from dislocation pile-ups with a small-angle scattering spectrometer of sufficiently high resolution.

A discussion of small-angle instrumentation was led by A. Franks, but, since the instrumentation is rather well developed in this field, little heat was generated in this discussion.

The conference was concluded with summarizing remarks by A. Guinier, and he stated that, since the meeting had been largely devoted to a single topic and since all the papers were in general agreement, his task was an easy one. The major accomplishment of the conference was the thorough understanding of the role of double Bragg scattering in studies of the small-angle scattering from metals. While the conclusion that double Bragg scattering can be put to little, if any, use was disappointing, it was stated by Guinier in his summary that "we are now able to make better use of this method (small-angle x-ray scattering) in the cases where it is still the best tool to study submicroscopical inhomogeneities". The host, Midwest Research Institute, is to be congratulated for conceiving and managing this successful conference. The proceedings of the conference will be published subsequently as a special section in the *Journal of Applied Physics*.

¹ Atkinson, H. H. and Hirsch, P. B., *Phil. Mag.* **3**, 213 (1958).