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ACTIVE MEASUREMENTS OF DEFECT PROCESSES IN
SHOCK COMPRESSED METALS AND OTHER SOLIDS¹

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Solid samples have been routinely recovered for examination after having been subjected to high pressure shock loading. Such investigations have revealed many unique and interesting defect features and are essential if a detailed understanding of shock deformation processes is to be achieved. Nevertheless, examination of samples hours or days after they are compressed for only a few microseconds in a loading whose rise time may be subnanosecond fails to address the relationship between the residual defect structure and that existing during the loading. Electrical probes, and to a lesser extent optical probes, have provided reasonably direct measurements of defect states and some limited information on the evolution of these states. For example, measurements of the electrical resistance of metals provide an indication of vacancy concentrations. Similarly, measurements of shock-induced electrical polarization in insulating solids have provided evidence that large numbers of point defects are generated and displaced by the stress and velocity gradients within the shock fronts. Optical measurements of shock-induced bleaching of color centers in NaCl have provided some evidence for kinetics of the formation of higher-order point defects. In this paper I review the status of active measurements which have provided information about shock-induced changes in the defect state of solids.

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I. INTRODUCTION

Despite the potential importance of point defects in shock-loaded solids it has proven difficult to obtain quantitative data on their concentrations and, indeed, there is considerable debate as to whether the concentrations are sufficient to cause significant effects. Nevertheless, evidence is accumulating that shock-induced defects, especially point defects, have a significant effect on compressional (1), mechanical (2), electrical (3), optical (3), thermodynamic (1), chemical (4-6), and structural processes (7,8) in shock-loaded solids. Evidence for high residual concentrations of point defects in shock-loaded metal samples preserved for subsequent examination has been summarized by Orava (9) and Leslie (10) and is found to consist of enhanced aging, inexplicably large strengths, unusually high densities of dislocation loops, jogs and dipoles and high densities of point defect clusters. Murr and coworkers (11) found evidence in transmission electron microscope observations on shock-loaded molybdenum of increases in vacancy concentrations of a few percent. Even though these residual effects may be readily apparent, examination of samples hours, days or months after an event in which the principal deformation is completed in a few nanoseconds, maintained for a few microseconds and complicated by a subsequent history including rapid unloading and possibly slower two or three dimensional plastic deformation, leaves open questions of the relation of the residual effects to those existing at various stages of the shock-loading event. Indeed, an overall study of the various probes of shock compressed matter assembled by Davison and Graham (3) leaves a strong impression that no single probe of material behavior, no matter how skillfully utilized or critically examined is sufficient, in itself, to support strong, comprehensive conclusions. The shock process is too complex and typical probes are too specific to warrant confident extrapolation of results to regimes remote from that under study.

It is my object in preparing this paper to review investigations which provide information on defect processes occurring while the sample is being subjected to shock loading; i.e. synchronous with the loading. Emphasis will be placed on point-defects, metals, and electrical probes, but work on other solids and on optical probes will be cited where appropriate. Some careful investigations of residual property measurements to the study of point defects in copper will first be considered after which active resistance measurements on shock-loaded metals will be summarized. The defect processes indicated by measurements of shock-induced electrical polarization in ionic crystals and polymers will then be commented upon. Finally, evidence from optical measurements and other studies indicating enhanced atomic migration in shocked solids will then be examined.

II. VACANCY CONCENTRATIONS IN COPPER FROM RESIDUAL RESISTANCE MEASUREMENTS

Two careful investigations of residual effects in shock-loaded copper, that of Brillhart, et al (12), and that of Mogilevskii (13), have produced evidence for significant increases in vacancy concentrations in samples subjected to controlled loading and preserved for subsequent study. The work of Brillhart, et al was carried out with samples shock loaded at room temperature while Mogilevskii's work was carried out with samples shock loaded at liquid nitrogen temperature.

Brillhart, et al (12) carried out detailed isochronal annealing studies over a temperature range of 298 to 675K, with measurements of density, hardness and resistivity. Stored energy release was also investigated. Their samples were cut from OFHC rolled and annealed copper plate and shock loaded for them at 7.5, 15, 25, 35 and 44 GPa by Dieter. Details of loading methods, pulse duration, and sample configuration are, unfortunately, not given, but the samples were driven into a water quenching bath to control post-shock sample temperature. They were kept in dry ice until test samples of 6 mm square cross section and 33 mm length were cut from the plates.

The annealing measurements indicated different characteristic behaviors in three temperature ranges. Although distinctive, systematic, behaviors were observed, conclusions based on the individual material probes were inconsistent with one another. For example, density annealing measurements indicated vacancy concentration of about 3×10^{-4} whereas resistivity measurements indicated vacancy concentrations of about an order of magnitude less. For these reasons the authors did not come to quantitative conclusions about vacancy concentrations even though significant concentrations were indicated. Since these samples were subjected to shock heating of hundreds of degrees, the residual vacancy concentrations could be expected to be lower than those initially present in the hot, shock-compressed sample.

Mogilevskii (13) has carried out a careful study of defects, including vacancies, interstitials and dislocations, on 99.996% pure copper wire samples shock loaded at liquid nitrogen temperature. The shocked samples were explosively ejected into a liquid nitrogen bath and stored in liquid nitrogen until electrical leads were attached. Isochronal annealing measurements of hardness and resistivity were carried out over the temperature range from 80 to 600K. Three samples had been explosively loaded at pressures of 7, 22 and 38 GPa in a system designed with spall elements to minimize deformation due to unloading. His interpretation of annealing measurements made on these samples is shown in Table I.

TABLE I. Defects in Shock-Loaded Copper
as Indicated by Resistivity Annealing^a
(After Mogilevskii, (13))

Pressure GPa	Vacancy Concentration	Interstitial Concentration	Dislocations m^{-2}
7	7×10^{-5}	5×10^{-5}	1×10^{15}
22	7×10^{-5}	5×10^{-5}	1×10^{15}
38	2×10^{-4}	6×10^{-5}	1.3×10^{15}

^aCalculation of the contribution of various defects to changes in resistivity upon annealing are based on the following identifications: 80-240K, interstitials; 240-340K, interstitial complexes; 340-450K, vacancies; > 450K, dislocations.

Mogilevskii's investigation indicates vacancy concentrations in the range of 10^{-4} . The low initial temperature of the samples minimized uncontrolled annealing and kept shock temperatures below room temperature except in the 38 GPa experiment. In the later case annealing of vacancies would be expected, hence, concentrations existing in the shocked stage would be greater than the reported peak value of 2×10^{-4} .

Mogilevskii's study minimized uncontrolled thermal annealing but leaves open questions of how point defects produced under cold deformation relate to point defects produced under higher temperature deformation and the relationship between residual concentrations and those existing in the shocked state. This later question must be addressed by active measurements; i.e. those carried out more or less synchronously with the shock loading.

III. VACANCY CONCENTRATIONS FROM ACTIVE RESISTANCE MEASUREMENTS

As indicated in Table II, numerous active resistance measurements have been made on shock-loaded metals. Most of the measurements shown in Table II were carried out to establish calibrations for piezoresistant stress gauges while others were taken to establish pressures at which structural transformations are initiated. Although only the works of Dick (14), and Dick and Styris (15) were carried out in sufficient detail to extract quantitative data on defect concentrations, evidence for excess resistivity, likely due to defects, is noted in every case where resistance measurements under shock loading are compared to those under high pressure hydrostatic compression. A recent study (16) of the widely investigated metal, Manganin, quantifies irreversible residual and incremental loading resistance effects. The work of Grady and coworkers (17, 18) adds an extra dimension concerning the relation of the defect-resistance after load release

to that observed while the sample is under load. Although the resistance measurements of Keeler (19) are not sufficient for detailed analysis, their resistivity values indicate an excess defect resistivity about equal to that seen in silver by Dick.

TABLE II Summary of Active Measurements
of Resistance in Metals^a

Sample	Pressure Range GPa	Investigations Number
Iron	2 to 150	5
Nickel	2 to 14	1
Copper	4 to 14	1
Lead	1.5 to 1.9	1
Cadmium	0.0 to 2.8	1
Indium	0.0 to 1.5	1
Bismuth	1.0 to 1.9	1
Manganin	0.2 to 30	Many
Ytterbium	0.1 to 22	4
Silver	2.5 to 12	1

^a See the summary in Davison and Graham (3).

The work of Dick (14) and Dick and Styris (15) on silver is sufficiently detailed to establish values for vacancy concentration in the samples while they were under shock loading. They studied the electrical resistivity of 15 to 25 μm thick silver foils over a stress range of 2.5 to 12 GPa. Materials of two characteristically different purities, as indicated by both resistivity measurements at liquid helium temperature and spectroscopic analysis, were investigated. The foil samples were carefully implanted in sapphire disks for mechanical impedance matching and shock loading was carried out with a compressed gas gun. After accounting for the resistivity change due to the shock-induced rise in temperature, and that due to isothermal, hydrostatic compression, an excess resistivity attributed principally to point defects, was obtained. This excess resistivity, as much as 17%, is shown as a function of stress in Figure 1. The two materials were found to exhibit somewhat different behaviors but both followed a defect resistivity which varied as the $3/2$ power of the compression. Peak vacancy concentrations of about 2×10^{-3} were observed at 12 GPa.

The study of Dick provides both quantitative data on point defect concentrations in the shocked state and a functional relation for the dependence of these concentrations on compression. By comparing the data on copper by Keller (19) to that of Dick, vacancy concentrations in copper of about 10^{-3} at 10 GPa are obtained.

FIGURE I. The contribution of defects to the resistivity of shock-loaded silver, $\Delta\rho_D$, is found to be strongly dependent on pressure in the works of Dick and coworker (14, 15). The data indicate a vacancy concentration of 2×10^{-3} at 12 GPa.

The active resistance measurements obtained by Grady and coworkers (17, 18) were carried out on 0.05 mm thick ytterbium foils etched in a grid pattern and encapsulated between 0.125 mm thick epoxy-fiberglass sheets. Measurements of the resistance of the samples were carried out while they were subjected to shock loading with impactors chosen to control pulse amplitude and duration and to provide a variety of unloading patterns. In experiments to a maximum stress of 600 MPa, resistance data for shock loading were compared to values expected for hydrostatic loading and those expected from elastic-plastic piezoresistive behavior. The shock

measurements showed a resistance change exceeding both hydrostatic and elastic-plastic response. When the residual resistance observed following unloading of the sample to zero stress was subtracted from the resistance measured under shock, the resulting resistance value is in agreement with the piezoresistive calculation.

The observations and analysis by Grady and coworkers provide evidence to support a defect state in ytterbium after weak shock unloading that is the same as that during shock loading. The active resistance measurements after unloading add a significant extra dimension to resistivity studies. Unfortunately, the time resolution of resistance measurements on metals is necessarily limited to the extent that values are typically recorded at least 50 to 100 nanoseconds after the initial shock deformation. Dielectric materials lend themselves to measurements within a few nanoseconds and may permit faster probing of defect processes.

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IV. DEFECT PROCESSES INDICATED BY SHOCK-INDUCED POLARIZATION IN IONIC CRYSTALS AND POLYMERS

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It has been known for many years that self-generated electrical signals appear in external circuits connected to dielectric samples subjected to shock loading (3, 20). If the samples are not piezoelectrics, ferroelectrics, or electrets these electrical signals are anomalous in terms of processes in thermodynamic equilibrium. The electrical configuration within the sample can be deduced from the recorded voltage waveform. In cases in which electrical polarizations in bulk are indicated, the material is said to exhibit shock-induced polarization. The magnitudes and wave shapes of polarization signals produced by many substances have been investigated and models have been developed to explain general features of shock-induced polarization in ionic crystals and polymers. These models are based on shock-induced generation of defects and their relative displacement in the velocity and stress gradients within the shock. Point defects are thought to be responsible for polarization in ionic crystals while for polymers the defects are thought to be radicals or other electrically charged molecular groups.

The cation-vacancy dipole model for shock-induced polarization in ionic crystals was developed by Mineev and coworkers in the Soviet Union (3, 20, 21) on the basis of an extensive and careful study of thirteen different ionic crystals with a wide range of ionic radii. The effect was investigated in various crystallographic orientations in a number of crystals. These Soviet scientists found that for compressions less than about 30% the magnitudes of the maximum shock-induced polarizations were the same for a given cation and independent of the anion. Those crystals with the smallest cation radius were found to generate the largest polarizations. Mineev

and coworkers have proposed that these polarization signals are the result of shock-induced formation of cation vacancies within the shock front and the subsequent displacement of the cation relative to the vacancy within the shock front. The resulting configuration creates cation-vacancy dipoles which, if present, in sufficient quantities, can cause a detectable bulk polarization. The magnitude of the polarization is controlled by the average component of dipole moment in the shock propagation direction and the number of dipoles in a unit volume of the crystal. The average component of dipole moment represents an average over the values of relative particle displacement and all dipole directions. Observed relaxations in polarizations are the result of relaxation of the point defects toward an equilibrium configuration.

From the magnitude of the polarization signals the average number of vacancies displaced to form dipoles can be determined on the basis of an estimate of their relative displacement. From estimates of vacancies produced by static plastic deformations, average displacements of 1 to 10 lattice constants are obtained. For a shock compression of about 10%, the average numbers of dipoles required to produce the observed polarizations for the displacement cited above is about 10^{24}m^{-3} . This represents an average dipole concentration of about 10^{-4} . If a low percentage of vacancies actually contribute to the average dipole concentration, which seems reasonable, vacancy concentrations of a few percent are indicated. The polarization measurements indicate that the point defects are generated and the displacement process is completed in times less than 10 nanoseconds.

A key feature of the Soviet cation-vacancy dipole model for shock-induced polarization is the relative defect displacement due to the stress and velocity gradients within the shock front. To date there has been no direct observation of such displacement (see a later section on diffusion) but independent evidence for such displacement is obtained in shock-induced polarization studies of polymers.

Although more feeble than those observed in ionic crystals, shock-induced polarization signals are readily observed in polymers. Graham (22) has recently proposed that shock-induced polarization in polymers is the result of mechanically-induced bond scission and the subsequent relative displacement of the electrically charged radicals within the shock front. Relative displacements of a few characteristic monomer sizes seem reasonable. This model is supported by evidence from independent mechanochemistry studies in polymers and correlations between observed polarization and monomer repeat units.

Considering their anomalous character, shock-induced polarizations in ionic crystals and polymers provide credible evidence

for processes involving defect generation and relative displacement in, or in the vicinity of, shock fronts. Furthermore, Dremin and Breusov (7) have proposed that such relative motion accounts for the rapid rates of structural transformation observed in shock-loaded solids. Nevertheless, electrical measurements and phase transformation rates provide only indirect indications and it is crucial to the understanding of defect processes to seek more direct evidence for the displacements proposed in the models. Optical probes appear to offer the potential for atomic scale observations.

V. OTHER PROBES OF DEFECT PROCESSES

Measurement of point defect concentrations and of the relative atomic displacement occurring while substances are under shock compression is clearly a difficult task but one within the capability of present or developing technology. Certain electrical probes have subnanosecond capability, but optical probes are required to observe processes in the 1 to 100 picosecond range. Available optical measurements demonstrate the potential for additional investigations by this method.

Studies of the effect of shock compression on color centers include the range of time scales extending from measurements of the properties of recovered samples to measurements taken while samples are under shock loading. The early work in this field is well summarized by Doran and Linde (23). They report an investigation in which an NaCl crystal colored by radiation was subjected to a 1.1 GPa shock loading pulse having a duration of about 1 microsecond. Examination of residual optical properties following the successful recovery of the sample showed the crystal was substantially bleached by the shock loading. This observation, and the further observation of enhanced bleaching rates, indicated shock-induced formation of a significant concentration of electron traps.

A more comprehensive investigation, at the much higher pressures of 10 and 30 GPa, was carried out by Lapshin, et al (24). They found that yellow-brown NaCl crystals were transformed to deep blue-violet after shock loading. The post-shock color is apparently due to the presence of colloidal metallic sodium in concentrations in the range of 10^{-4} to a few times 10^{-3} .

Although the residual effects observed in the optical investigations described above provide evidence for significant levels of point defects they suffer from the uncertainty of other post-test observations as to their relation to defect processes occurring while the samples are under the action of the shock-loading. The picture can only be completed by active measurements.

Yakusheva, et al (26) have carried out active measurements of color center bleaching of NaCl crystals under plane-wave shock loading of 3 and 18 GPa. They observed that the color of the crystal changed from yellow to white upon entry of the shock. At the lower pressure this observation cannot be explained by an increase in temperature or shift in the absorption band, and so provides strong evidence for shock-induced generation of electron traps in the vicinity of the shock front. Gross kinetics of the process are also indicated by the observed recoloration of the crystal after 500 nanoseconds.

Kormer (27) has reported observations of optical absorption of NaCl while under very high pressure shock loading which, when combined with resistance measurements, indicate the presence of electron donor densities in concentrations of a few $\times 10^{-3}$. Those measurements were carried out on samples compressed to such high pressure and temperature that it is difficult to interpret them in terms of well-known defect processes. Kormer has also reported, from evidence obtained in optical measurements, that the structural transformations in shock-loaded KCl and KBr are completed in 10 picoseconds.

Active measurements of optical properties have also been used to look for direct evidence for relative atomic displacement in shock fronts. Yakusheva, et al observed the reflectivity of a 100 Å thick reflecting surface placed on Plexiglass and LiF samples subjected to shock loading of about 10 GPa. It was expected that the displacement of atoms from the host into the reflector would change the reflectivity if such effects were significant. No effect was observed.

Measurements of mass diffusion taken on solid samples recovered from shock loading show evidence for diffusion over distances of tens of micrometres corresponding to diffusion coefficients perhaps three orders of magnitude greater than those of the starting samples (29-31). These large diffusion distances do not seem attributable to processes taking place directly in shock fronts, but are probably due to diffusion in the hot, highly defective sample that has returned to atmospheric pressure after shock loading.

VI. SUMMARY

In the present review I have summarized results from some principal investigations of shock-induced point defects in solids. The combination of residual and active measurements is found to provide evidence for typical vacancy concentrations of from 10^{-5} to 10^{-2} . Although the results of a single investigation are not definitive, the collective picture presented by the various investigations is one in which shock-induced increases in vacancy

6-211 x 10⁻⁷ in. in.

Line typewriter

10% reduction

35-111 x 61 phone wiring etc

20 x 10⁻⁷ plus 111 in. in.

concentration are large enough to have a significant effect on many physical and chemical processes. Certainly, more detailed study of point defects is called for. It appears that measurements obtained from a variety of different probes, both residual and active, on a single material would provide the most effective basis for developing a detailed quantitative picture of point defects in shock-loaded solids.

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