Table 2. SUMMARY OF PALÆOMAGNETIC DATA FOR CRETACEOUS AND TERTIARY ROCKS OF NORTH AMERICA

Formation	Age	Collection site		*D*	*I*	*N*	*S*	α_{95}	*K*	Pole position		dχ	dψ
		Lat.	Long.							Lat.	Long.		
Dakota sandstone †	Cretaceous	34 N.	110 W.	159·0	−60·5	3	9	—	—	72·5 N.	169·5 W.	—	—
Isachsen diabase	Cretaceous ?	79 N.	104 W.	283·5	+79·5	20	40	7·5	20	69 N.	180 W.	14·1	13·6
Basic intrusives Mount Megantic	Cretaceous	45·5 N.	71 W.	157·0	−52·0	12	24	6·5	44	68·5 N.	171·5 E.	8·9	6·1
Canadian basalts *	Late Tertiary	61 N.	134·5 W.	348·5	+75	46	—	4	28	85 N.	145 E.	7·2	6·7
Payette *	Miocene Plocene (?)	43 N.	115·5 W.	1·5	+62·5	13	—	2·5	258	88·5 N.	60 W.	4	3
Columbia River basalts *	Miocene	46·5 N.	120 W.	3·5	+69·5	73	—	8	4	83 N.	105 W.	13·5	11·5
Ellensburg *	Late Miocene Early Pliocene	46·5 N.	120·5 W.	0·5	+68·5	23	—	9	12	85 N.	115 W.	15·5	13
Duchesne River *	Tertiary	40·5 N.	110 W.	2	+65	24	85	5	14	83 N.	99 W.	8	6
Laney Shale *	Eocene	41·5 N.	109·5 W.	353·5	+62·5	19	—	6	30	85 N.	170 W.	9·5	7·5
Green River *	Eocene	39·5 N.	108 W.	345·5	+65·0	7	—	4·5	168	77·5 N.	158 W.	7	6
Wasatch *	Eocene	44·5 N.	109 W.	351·5	+63·5	4	—	17	30	84·5 N.	180 E.	26·5	21

N, number of samples; *S*, number of specimens; *α*, radius of the circle of confidence at the 95 per cent level of probability; *K*, estimate of the precision parameter; *dχ*, *dψ*, semi-axes of the oval of confidence; *, as reported by Cox and Doell (ref. 7); †, as calculated by us from the means of the three reversely polarized samples reported by Runcorn (ref. 4).

measurements of the Dakota Sandstone[4]. Runcorn considered that only the three reversely polarized samples of the six measured by him were magnetically stable. The mean magnetization direction was recomputed by us for these three samples, the mean of the first sample being based on four specimens, that of the second one on three specimens, and that of the third on two specimens. The recomputed mean direction for this group and the corresponding pole position are given in Table 2. The second Cretaceous pole available in the literature is based on the magnetization measurements of 12 samples[5] from the magnetically stable basic intrusive rocks of Mount Megantic. The age of these rocks has been established as Cretaceous by potassium/argon age determination[6].

The proximity of the poles derived from the Isachsen diabase and the Mount Megantic rocks, the magnetic stability of the rocks in both cases, and the general departure of these two poles and of the pole derived from the Dakota Sandstone from the Tertiary poles all indicate that the Isachsen diabase rocks are probably of Cretaceous age. Furthermore, this interpretation is not in contradiction with the date postulated by the available geological field evidence.

We thank the Director of the Geological Survey of Canada for allowing publication of this article and Mr. J. Babbin, of the Polar Continental Shelf Project, for collecting the Isachsen diabase samples.

Note added in proof. Since the manuscript for this article was first submitted, potassium/argon (whole rock) analyses were made for samples from sites 1 and 10. The ages obtained were 241 and 249 m.y. respectively, which would set the age of the dykes as Permian. This indication is fully incompatible with the biostratigraphic and palæomagnetic data (100 ± 25 m.y.) and no reasonable explanation has been found so far for this incompatibility.

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DECOMPOSITION OF SINGLE CRYSTALS OF LEAD HYDROXIDE IN THE ELECTRON MICROSCOPE

By DR. M. J. SOLE* and DR. A. D. YOFFE

Physics and Chemistry of Solids, Cavendish Laboratory, University of Cambridge

IN recent years there have been a number of investigations into the way in which solids decompose in the highly ionizing beam of the electron microscope. Compounds which have been examined include silver azide[1], magnesium hydroxide[2], silver iodide[3], lead iodide[4] and silver cyanamide[5]. This article describes some observations on the behaviour of lead hydroxide. This compound decomposes in an interesting manner with the formation of both crystallographic cavities and crystallographic nuclei of decomposition product.

The investigation arose out of a more general investigation of the structure and stability of lead and other inorganic cyanamides[6]. During the preparation of single crystals of lead cyanamide for examination in the electron microscope crystals of lead hydroxide are also formed. The latter are particularly suited for examination by transmission electron microscopy and by electron diffraction.

Preparation of Crystals

The preparation of crystals involved slow precipitation in alkaline solution. This was achieved by allowing ammonia gas to diffuse through a layer of ether covering a solution of lead cyanamide in dilute acetic acid. Before adding the ether, an evaporated carbon film was floated on to the surface of the acid solution and gold electron microscope grids (500 mesh) were placed in the solution on platinum gauze supports. Ether was then added to a depth of about 5 mm. The carbon film remained at the interface, though some of its coherence was lost. The vessel and its contents were then placed in a large desiccator containing a beaker of 880 ammonia. After 15–30 min a yellow precipitate of lead cyanamide was clearly visible and the vessel was then removed. Some crystals (not visible) are formed on the carbon film and this was then caught up over the grids by raising the support gauze through the film. Most of the liquid adhering to the grids was removed with filter paper, and, after drying, the grids were ready for examination in the microscope. This was a Siemens-Elmiskop I electron microscope operated at 80 kV.

Crystals of both lead cyanamide and lead hydroxide are to be found on the carbon support film. The former were identified by their electron diffraction pattern[7].

The crystals of lead hydroxide were clearly distinguishable both by their appearance and by their hexagonal diffraction patterns, which showed them to be in the (0001) orientation[8]. They were generally sufficiently thin for extinction contours to be visible and were hexagonal in shape (though the hexagons were seldom complete). Because of a strong tendency towards overgrowth, Moiré patterns were frequently observed, but dislocations were rarely seen. The possibility that the crystals were a basic lead salt seems unlikely as crystals with the same

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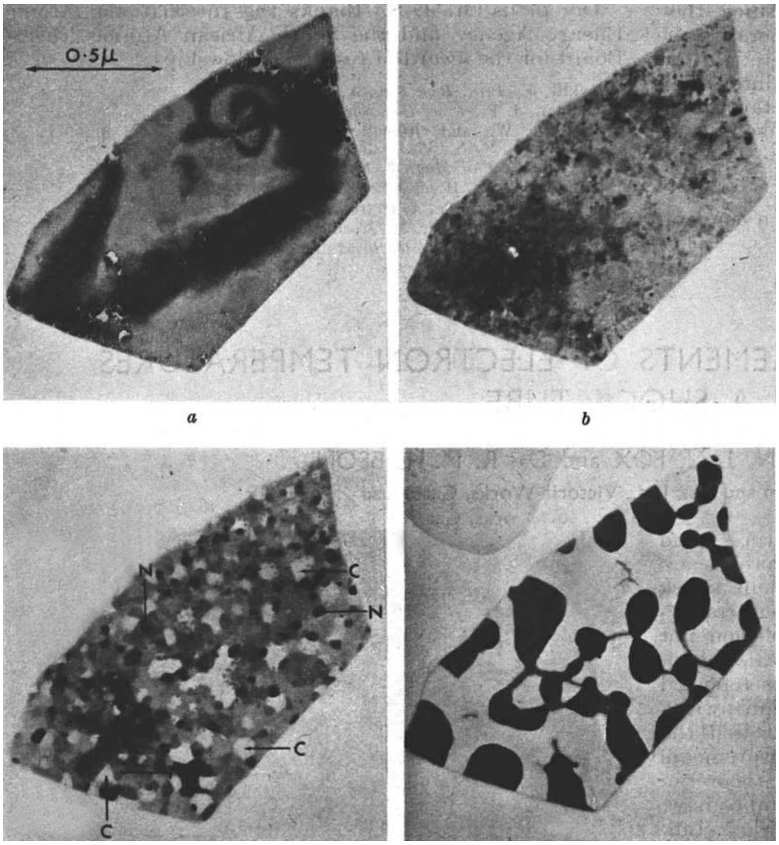
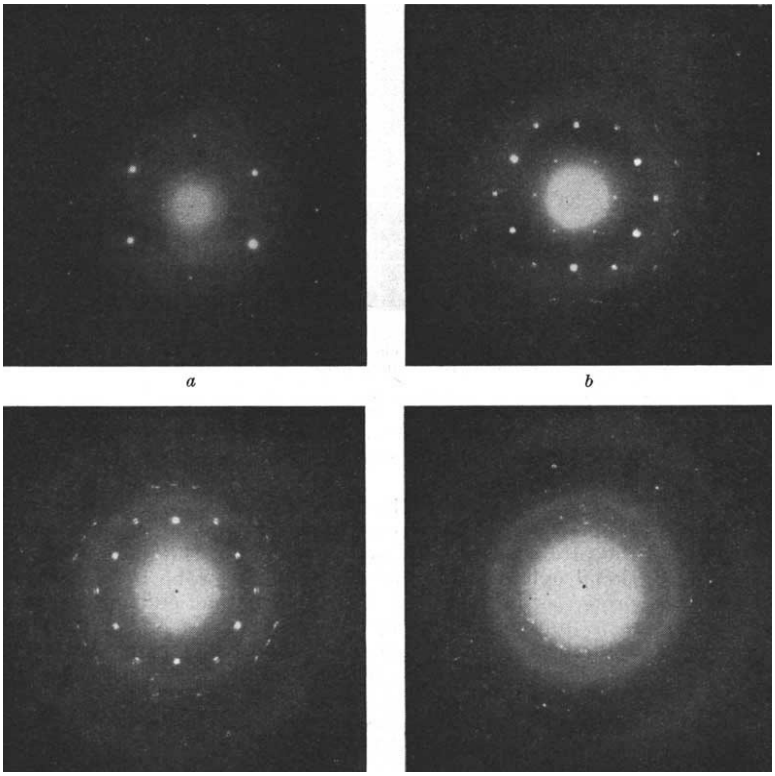
* Present address: Metallurgy Division, Atomic Energy Research Establishment, Harwell.



a *b*

Fig. 1. A sequence of electron micrographs of a single crystal of lead hydroxide decomposing in the electron beam. Some of the crystallographic cavities (*C*) and crystallographic nuclei of decomposition product (*N*) are arrowed



a *b*

Fig. 2. Electron diffraction patterns of the same crystal as in Fig. 1 and corresponding to similar stages of decomposition

appearance and diffraction pattern could be prepared from acid solutions of both lead acetate and lead nitrate.

Behaviour in the Electron Beam

The lead hydroxide crystals were fairly stable in the electron beam under normal operating conditions but decomposed as the beam current was increased. Fig. 1*a* shows the appearance of a single crystal containing a few flaws and cracks. Some slight localized decomposition has occurred in their vicinity and along edges. Fig. 1*b* shows the same crystal after some overall decomposition has occurred. It should be noted that now some of the 'holes' previously visible have disappeared, and this may be taken as evidence for the mobility of the products of decomposition. The crystal is now mottled in appearance and there are some discrete nuclei of a decomposition product, presumably metallic lead. The Bragg extinction contours have disappeared. The next stage in the decomposition is particularly worthy of note, being characterized by the formation of crystallographic voids and nuclei as shown in Fig. 1*c*. These have a tendency to be triangular or hexagonal in shape as indicated by the arrows. At even higher beam currents decomposition to metallic lead is complete. The metal is highly mobile and aggregates as shown in Fig. 1*d*. These aggregates are always confined to the region originally occupied by the crystal and they exhibit contrast effects which may be thickness fringes or due to stacking faults.

The corresponding diffraction patterns are shown in Fig. 2. As decomposition proceeds the spots elongate and become double. Prior to the final stage of decomposition lead hydroxide is still present with the same lattice spacing ($a = 5{\cdot}24$ Å), while the second series of spots has moved progressively inwards, the *a* spacing increasing to about 5·50 Å. There are also additional spots due to randomly orientated lead and after complete decomposition only these remain.

Discussion

It would appear from the diffraction patterns that the decomposition of lead hydroxide to metallic lead and gaseous products involves the formation of an intermediate which is probably an oxide of lead. This intermediate is oriented with respect to the parent lattice and the crystallographic nuclei of Fig. 1*c* may be ascribed to it. The lead, shown as black specks in Figs. 1*b* and 1*c*, seems to be more randomly oriented. The regions of light contrast which have crystallographic shapes may be attributed to voids or cavities within the crystal as in the case of lead iodide[4]. These cavities do not necessarily penetrate right through the thickness of the crystal. There are indications that the cavities enhance nucleation as in lead iodide, but they do not now have the same degree of mobility. Similar behaviour has also been reported for silver cyanamide thermally decomposed in oxygen[5].

These observations have been reported since they indicate that these phenomena are perhaps general to the decomposition of layer structures in which there is a fair degree of covalent bonding. It would be of interest to pursue the possibility that bonding along certain directions has a direct influence on the course of solid-state reactions of the general type:

$$\text{solid } A \rightarrow \text{solid } B + \text{gas } C$$

Electron microscopy and diffraction provide a convenient way for such an examination.

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DOUBLE PROBE MEASUREMENTS OF ELECTRON TEMPERATURES IN A SHOCK TUBE

By T. I. McLAREN, J. N. FOX and DR. R. M. HOBSON
Clarke, Chapman and Co., Ltd., Victoria Works, Gateshead

MEASUREMENTS of electron temperatures and concentrations by means of Langmuir probes are particularly attractive in studying the transient shock generated plasmas in shock tubes as, with proper arrangement, they provide spatial and temporal resolution not available from other techniques such as optical, microwave, expelled flux, etc. However, the results reported so far by, among others, Lin, Resler and Kantrowitz[1], Hollyer[2], Hand and Kistiakowsky[3], have indicated the difficulties of obtaining reproducible probe measurements which agree to within a reasonable factor of the theoretically predicted parameters. Furthermore, attempts have been made by Sakuntala, Von Engel and Fowler[4], Lin, Resler and Kantrowitz[1], Pain and Smy[5], Nagamatsu and Sheer[6], to measure the conductivity of partially ionized plasmas flowing through magnetic fields by the application of what is effectively a double-probe technique. Inconsistencies in these measurements are not surprising in view of the previous results in which the probe collection mechanisms were not complicated by the presence of a magnetic field.

The measurements reported here form part of the initial diagnostics in a programme to examine the interaction of partially ionized plasmas with the magnetic fields through which they are flowing. The physics of these processes will at a later stage be applied to the problems of engineering design in magneto-hydrodynamic power generators.

The electrically driven 2-cm shock tube in which these experiments have been carried out has been previously described by Hobson and McLaren[7]. The double probe technique of Johnston and Malter[8] has been adapted as indicated in Fig. 1 to the transient measurement requirements of the shock tube.

Initial experiments using copper, tungsten and gold probes outlined the difficulty of obtaining reproducible signal amplitudes from subsequent identical experimental conditions and this is indicated in Fig. 2. This attenuation in signal was attributed to alterations in the surface conditions of the probe at successive shocks. Mechanical cleaning, chemical etching, and glow-discharge cleaning between experiments produced no significant consistency and in many cases led to appreciable time delays between experiments in order to ensure initially similar clean vacuum conditions.

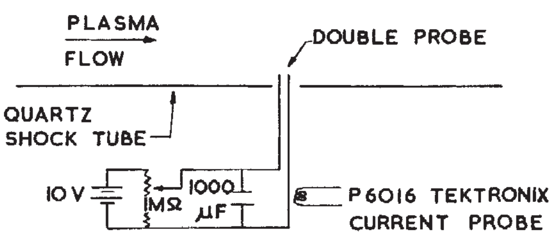

Fig. 1. Basic double-probe circuit

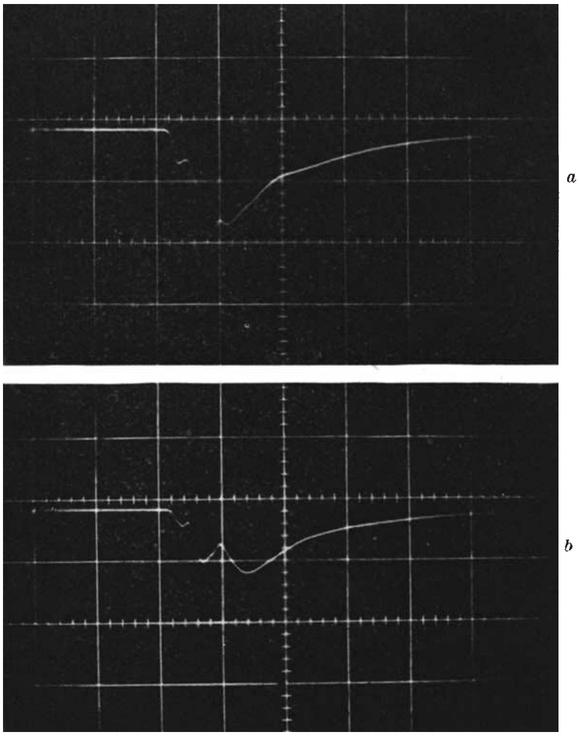

Fig. 2. *a*, Response of a gold double plane probe. *b*, Response of same probe in next shock. No cleaning between shots. Time-scale 20 μsec/major division; current scale 0·5 amp/major division

It was apparent that a more effective technique of probe-cleaning in vacuum was essential, and to this end the probe geometry was changed from the more conventional cylindrical, spherical or plane probe to a 'hairpin probe' as indicated in Fig. 3. In practice each hairpin is a unipotential surface when used as an element of a double probe, but can be electrically connected outside the vacuum system to enable a cleaning current to be passed through the probe wire between shocks. In general the probe is constructed from 0·008-in. tungsten wire and between shocks is flashed to white heat for a period of about 30 sec with continuous vacuum pumping at a pressure of about 10^{-5} mm mercury. Experiments with this new arrangement give a high degree of consistency and this is clearly seen in Fig. 4.