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Experimental modification of naturally deformed galena crystals and their grain boundaries

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SUMMARY. It is now well established that naturally-occurring sulphides—particularly galena—are frequently found in the deformed state and that at least part of this deformation can be eliminated by annealing in the laboratory.

The present contribution is concerned principally with galena, for which it is indicated: that tectonic deformation leads to natural work hardening, which may be reduced by simple heating *in vacuo*; that the rate of softening is related to the annealing temperature; that softening is related to recovery at low temperatures and to both recovery and recrystallization above 250–300 °C; that single crystals of galena exhibiting minor slip and kinking recrystallize yielding new, unstrained grains elongated parallel to the old slips and kinks, i.e. a foliation involving unstrained grains may form under a regime devoid of compressive stress; and that where deformed galena and deformed sphalerite coexist, the galena may be made to recrystallize completely (e.g. at 325 °C in 50 days) while leaving the deformation in the sphalerite entirely intact; apparently the coexistence of a strained component A and an unstrained component B in a deformed ore or other metamorphic rock does not necessarily indicate differences in age or deformational history—it may simply indicate that post-deformational temperatures have exceeded the recrystallization temperature of one component but not of the other.

THIS contribution reports some of the results obtained from some simple heating experiments on galena. The experiments were carried out in evacuated, sealed glass tubes, i.e. under the vapour pressure of the material involved, and were concerned with various phenomena associated with the recovery and recrystallization of deformed galena. Most of the experiments have been carried out on material in which deformation is conspicuous; some, however, have involved specimens in which deformation was not clearly apparent.

Fig. 1 shows a polished surface of a hand specimen of the material involved in most of the experiments—a highly coherent galena–sphalerite–garnet gneiss from the southern end of the Broken Hill orebody, New South Wales. The galena is the major constituent in which are set numerous augen of sphalerite and a few augen of garnet.

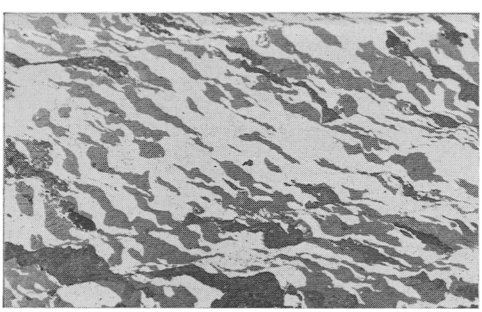


FIG. 1. Galena–sphalerite–garnet gneiss; galena nearly white, sphalerite medium grey, garnet dark grey to black. $\times 1{\cdot}5$.

Small 'slugs' of this material (approximately $10 \times 5 \times 4$ cm) were cut, polished, and etched. In each case grain structures were noted and photographed and 20 (in one case 70) microhardness measurements made on areas that could readily be relocated with great accuracy for succeeding hardness measurements and microphotography. Four such slugs gave initial mean Vickers hardnesses of 84·2, 83·2, 81·8, and 79·4. Each was then subjected to a series of isothermal heating runs at 200, 300, 400, and 500 °C; the results are shown in fig. 2. The galena softened, the rate of softening was related to temperature, and there was a general (though not invariable) increase in total softening with increase in temperature.

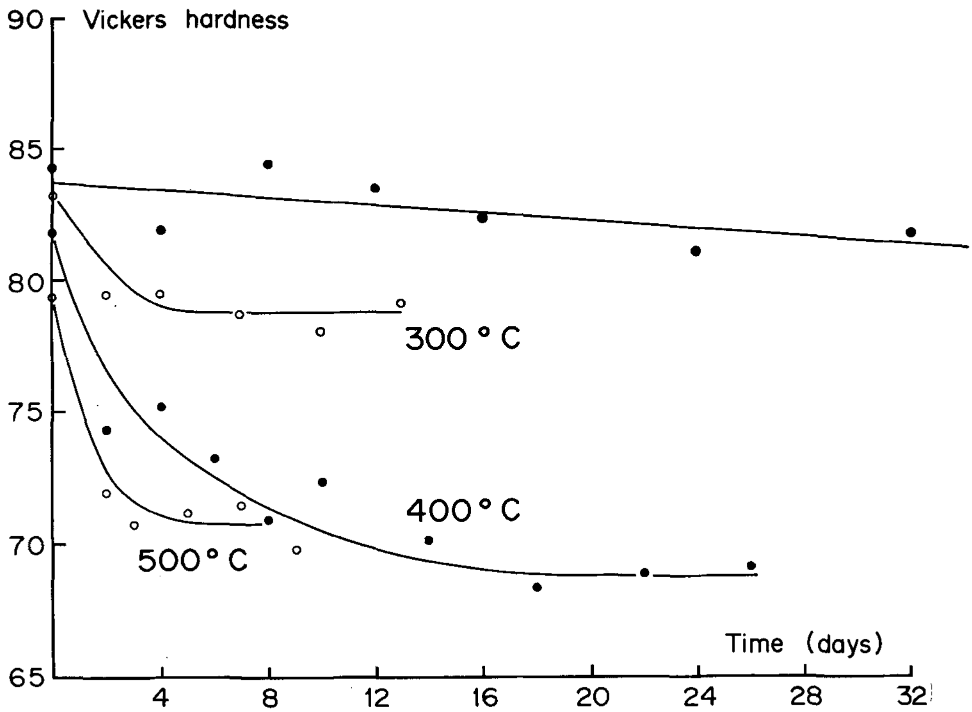
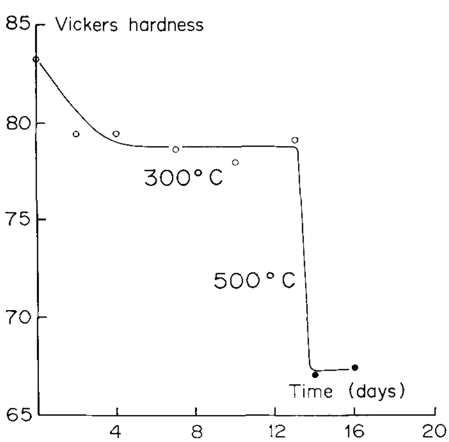


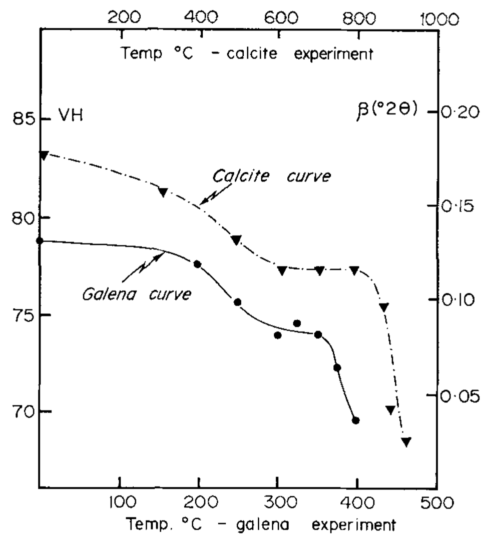
FIG. 2. Effect of heating on the Vickers hardness of deformed galena.

Fig. 3 confirms the relationship between heating temperature and degree of softening; the diagram involves a single slug (the one involved in the 300 °C curve in fig. 2), the open circles representing runs at 300 °C, the filled ones representing runs at 500 °C. The hardness at 300 °C had apparently stabilized after 4 or 5 days, but heating at 500 °C immediately—and dramatically—reduced it again.

It was noted in the experiments of fig. 2 that at 200 and 300 °C recrystallization was not evident, but that at 400 °C and particularly at 500 °C it soon became quite conspicuous. This suggested that softening might be due to both recovery and recrystallization. It may be mentioned that slight deformation of a crystal leads to changes in some of the physical properties of that crystal: e.g. it leads to increases in hardness, electrical resistivity, and breadth of X-ray lines. Appropriate heating leads to 'recovery', which involves the resumption of the hardness, resistivity, and X-ray line breadth possessed prior to deformation.

Gross (1965) experimentally deformed and then heated natural polycrystalline calcite; X-ray line breadth was measured after deformation and then again following each of a series of heatings carried out at successively higher temperatures, each heating lasting 1 h. The X-ray line breadth (β, in $°2\theta$) underwent an initial decrease, corresponding to recovery, followed by a pause and then a rapid decrease corresponding to recrystallization. This is shown in fig. 4, the calcite curve here representing the behaviour of material initially deformed by 5·6 %.





FIGS. 3 and 4: Fig. 3 (left), effect of heating on the Vickers hardness of deformed galena. Fig. 4 (right), effect of heating on the Vickers hardness of deformed galena compared with the effect of heating on the X-ray line breadth of deformed calcite.

Also shown in fig. 4 is the behaviour of our naturally deformed Broken Hill galena—in this case involving change in Vickers hardness rather than in X-ray line breadth. Temperatures used were lower than those used by Gross for calcite, and heating time at each temperature was 24 h. The analogy between the two phenomena in the two materials is clear. This shows, by again emphasizing the over-all analogy with metals, that from the physical point of view we are dealing simply with 'crystalline materials' rather than specialized categories of 'rocks', 'ores', 'ceramics', and so on.

Fig. 5 gives an abbreviated pictorial representation of the events involved in the latter half of the galena curve of fig. 4. Microphotograph 1 shows the ore in essentially its original state. Microphotograph 2 is taken after the 350 °C heat; recrystallization and grain growth is now conspicuous in the lower parts of the galena area. Microphotograph 3 follows heating at 375 °C and shows further recrystallization and grain growth, and microphotograph 4 (400 °C) shows recrystallization of the galena virtually complete.

While the galena of this experiment was quite obviously deformed, it was suspected,

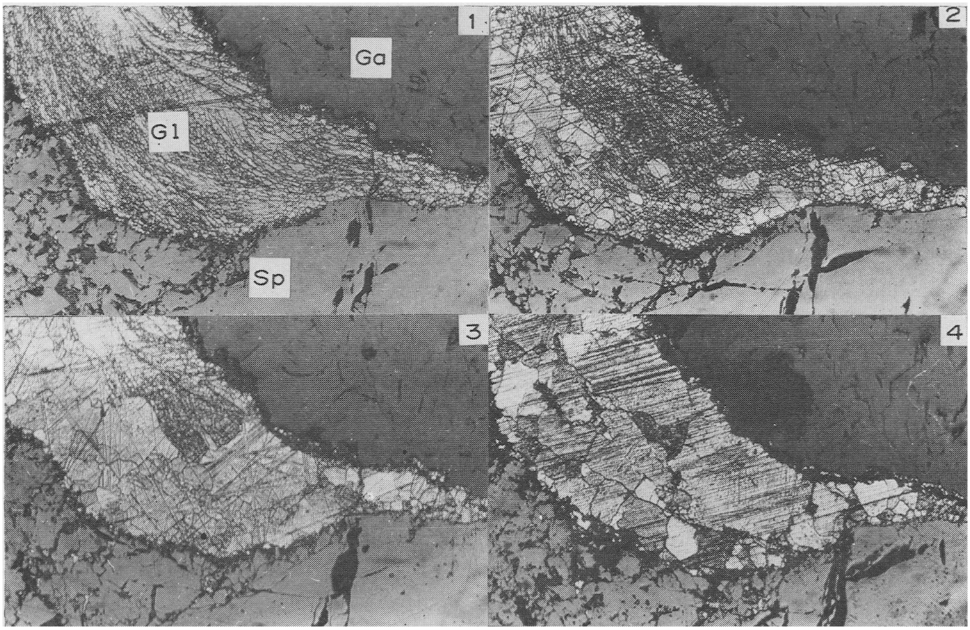


FIG. 5. Deformed galena after heating at various temperatures. Gl, galena; Sp, sphalerite; Ga, garnet. ×66.

from some associated work, that *some* galenas showing no obvious sign of deformation might in fact be slightly strained. Material described by others as 'massive and undeformed' from Broken Hill and Mount Isa was therefore taken and subjected to a few 2-day runs at 500 and 400 °C respectively. Fig. 6 shows the results. This indicates that numerous hardness measurements carried out to date in various laboratories may have determined deformation-induced hardnesses (i.e. natural 'work-hardening') rather than hardnesses that were intrinsic properties of the minerals concerned and that hardness measurements combined with simple heating may constitute extremely delicate indicators of cryptic deformation.

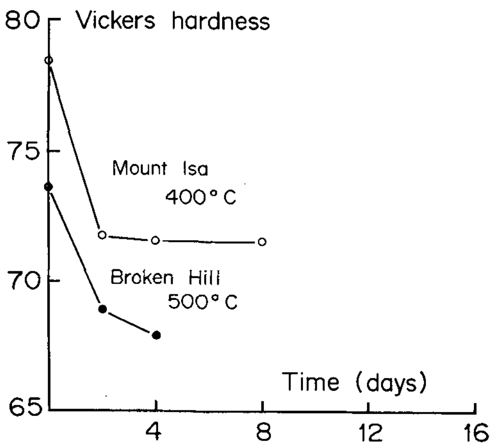


FIG. 6. Effect of heating on the Vickers hardness of two supposedly undeformed galenas.

In a related experiment, the behaviour of sphalerite during the recrystallization of accompanying galena was studied very carefully. Comparative behaviour of the two minerals as a coexisting pair is illustrated by fig. 7. Microphotograph 1 shows the original material lightly etched with HCl and thiourea; the etch shows the galena as highly deformed and finely granular, and the sphalerite to be kinked, twinned, and bent. Bending is very obvious, and the lower sphalerite area is distinctly splayed. Microphotograph 2 was taken after 14 days at 325 °C; the etch has not taken well on the sphalerite, but the development of enlarged grains of galena along the lower galena–sphalerite boundary is clear. Microphotograph 3, taken after a further 16 days at 325 °C, shows the galena almost completely re-textured as a result of recrystallization and grain growth; the sphalerite, however, appears to have retained all of its original deformational features. Under the simple conditions of this experiment, at a 'lower greenschist grade' temperature and in only 30 days the original deformation texture of the galena has been almost completely eliminated—but the original texture of the sphalerite has been left unaffected.

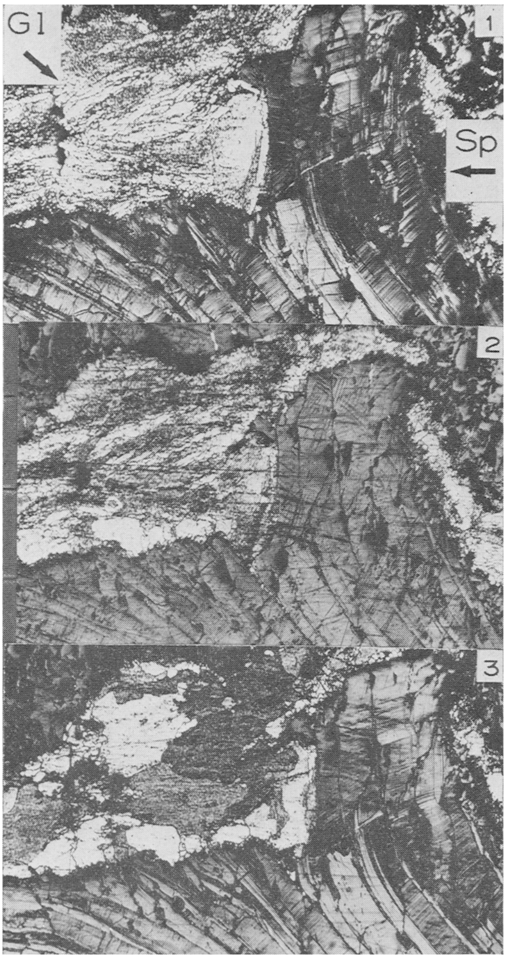


FIG. 7. Recrystallization of galena on heating, the accompanying sphalerite remaining unaffected. ×75.

Clearly therefore the coexistence of strained and unstrained components in a rock does not necessarily indicate different deformational histories and hence different ages. It may simply indicate that post-deformational conditions (particularly temperature) have exceeded those required for the recrystallization of some component(s), but not those required for the recrystallization of the other component(s).

That simple microscopical observation indicated that this was *probably* so was indicated by the author to Professor K. C. Dunham in conversation in 1964; the recent experiments establish the principle beyond doubt.

Recrystallization in metals, etc., may lead to the development of simple random aggregates, or it may lead to crystal structure or dimensional preferred orientations, or both. That some preferred orientations of minerals in rocks (including ores) may be due to annealing following deformation rather than simply to the deformation itself must therefore be kept in mind. Fig. 8 shows recrystallization of portion of a strained *single crystal* of galena. The clear white grains (showing particularly conspicuous scratches) of the central and lower and upper right-hand portions of the microphotograph 1 are new grains resulting from experimental annealing at 400 °C for 23 days. The light- to medium-grey areas are original low-intensity kink bands induced in the crystal by tectonic stress. The new grains are showing a distinct tendency to grow along the original kinks, and hence to develop an elongated form. Microphotograph 2 shows the same material after 25 days, following a rapid acceleration in recrystallization, with the formation of large numbers of new, flat grains. No compressional force was exerted during the experiment—the specimen simply lay under

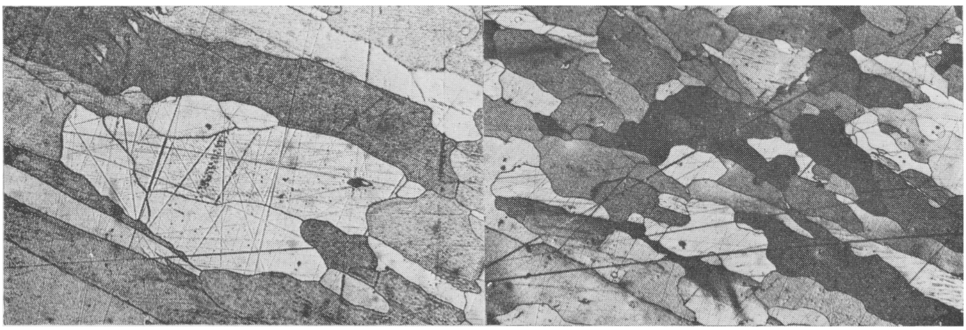


FIG. 8. Recrystallization of a strained single crystal of galena after heating at 400 °C. Fig. 8. 1 (left), after 23 days (×70); fig. 8. 2 (right), after 25 days (×23).

its own vapour pressure in the tube. We thus see that an apparent foliation—a 'growth foliation'—may develop in the absence of a deforming force. The platy grains so formed are, of course, themselves devoid of significant deformation. The possible significance of this in metamorphic studies is clear. For sulphide ores in particular orthodox interpretation would hold that the occurrence of *undeformed* sulphide grains in foliated arrangement indicated that the sulphide must have been deposited after deformation as a replacement of an earlier mineral that had been involved in a preceding deformational episode. Apparently this is not necessarily so.

These and a number of other experimental results obtained by the writer seem to indicate that much awaits to be learned concerning sulphide textures, and that studies of the highly mobile, essentially cubic and structurally simple sulphides may have hitherto unsuspected value in the understanding of metamorphic microstructures in general.

REFERENCE

GROSS (K. A.), 1965. *Phil. Mag.*, ser. 8, **12,** 801–13 [M.A. **18**-286].

[*Manuscript received 11 May 1970*]