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O T T A W A May 12th, 1944.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1645.

Examination of Two 40-mm. Brass Cartridge Cases.

(Copy No. 10.)

Bureau of Mines
Division of Metallic
Minerals.

Ore Dressing
and Metallurgical
Laboratories

CANADA

DEPARTMENT
OF
MINES AND RESOURCES

Mines and Geology Branch

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Origin of Material and Object of Investigation:

Under date of March 25th, 1944, Analysis Requisition O.T. 4159 was received from the Inspection Board of the United Kingdom and Canada, Ottawa, Ontario, requesting a metallurgical examination of two 40-mm. brass cartridge cases which were received on the same date. In a covering letter (File No. 10444, Index 1), of the same date, it was reported that the two cases were unduly soft in the region of one-half to two inches from the base.

In the letter it was further reported that, after working, the cases are subjected to a stress-relief anneal of 540° F. for one hour and that, in the past, this treatment has not affected the hardness of the cases. An examination of the cases to determine the cause of the softness was requested.

All results were submitted to the I.B.U.K. & C. verbally, on April 25th, 1944, but for purposes of confirmation are given here.

Examination of the markings on their bases showed that the cartridges had been manufactured in 1944 and that one was from Lot No. 79 and one from Lot No. 82. These numbers have been retained in this report in referring to the cases.

Hardness Tests:

Hardness tests were made along the length of the case on the outside, the cross-section, and the inside of the wall, using the Vickers hardness tester with a 10-kilogram load. For the determinations on the outside the whole case was held in a vise without internal support, while for those on the cross-section and inside, a longitudinal strip about ¼ inch wide was cut from each case and clamped in the vise. The zero point in measuring the distance of the hardness readings from the base was taken as the rim of the extractor groove, shown as the point A in Figures 1 and 2. The results of the hardness tests are tabulated in Table I and are shown graphically in Figures 1 and 2.

TABLE I.

Distance from A, inches	VICKERS HARDNESS NUMBER (10-kg. load)					
	Lot No. 79			Lot No. 82		
	Outer surface	Cross-section	Inner surface	Outer surface	Cross-section	Inner surface
¼	126	166		144	185	
½	125	153		150	171	
¾	117	146	113	122	162	123
1	117	142	115	122	149	124
1¼	141	161	138	150	161	132
1½	157	170	155	165	177	150
1¾	155	170	154	167	178	156
2	161	167	154	165	183	162
2½	159	180	158	163	182	166
3	156	173	160	160	176	166
3½	150	165	151	153	172	156
4	141	151	134	147	164	154
5	123	135	126	128	140	129
6	119	133	125	122	137	127
7	118	131	126	120	138	126
8	116	136	122	121	142	127
9	122	142	123	122	138	127
10	128	146	127	121	157	138
11	99	112	99.8	91	131	90.4
11½	79	94.5	85.8	78	104	88.8

Heat Treatment:

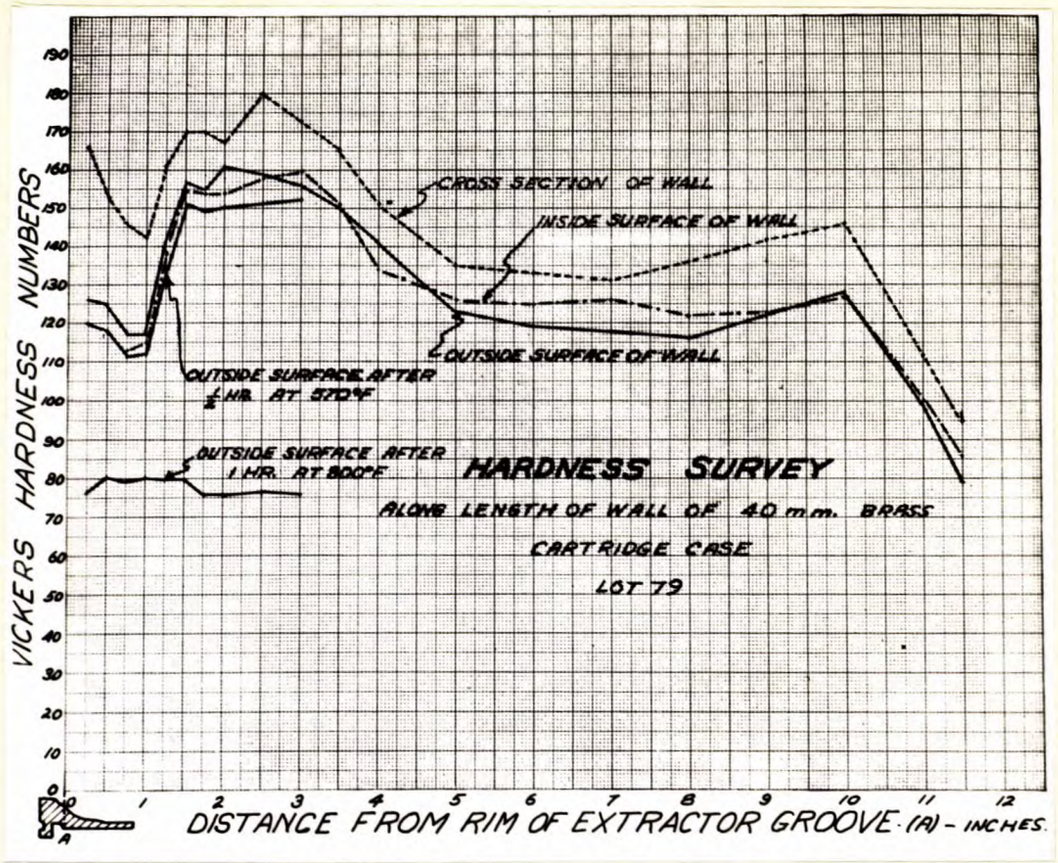
In order to obtain some material which was known to be partially recrystallized, for comparison in the X-ray diffraction experiments to be described later, a strip containing part of the base and the lower four inches of the wall was cut from each case. To obtain a piece which had just started to recrystallize, the strip from Lot No. 79 was annealed for ½ hour at 570° F. and hardness determinations were made along its length on the outer wall surface. To obtain a piece completely recrystallized, the strip was re-annealed at 800° F. for 1 hour and the hardness determinations were made again. The strip from Lot No. 82 was annealed for ½ hour at 600° F. and hardness determinations made along its length. The results of the hardness tests are tabulated in Table II and also are included in Figures 1 and 2.

TABLE II.

Distance from A, inches	VICKERS HARDNESS NUMBER (10-kg. load) Lot No. 79 570° F., ½ hr.	Lot No. 79 800° F., 1 hr.	Lot No. 82 600° F., ½ hr.
¼	120	75.4	104
½	118	80.8	102
¾	111	79.5	104
1	112	80.2	104
1¼	132	79.8	105
1½	151	79.5	125
1¾	149	75.7	129
2	150	76.0	134
2½	151	76.3	144
3	152	75.7	142

(Figures 1 and 2 follow, on Page 4.)
(Text continues on Page 5.)

Figure 1.



VARIATION OF HARDNESS OVER LENGTH OF CASE, LOT NO. 79.
Note drop in hardness near base.

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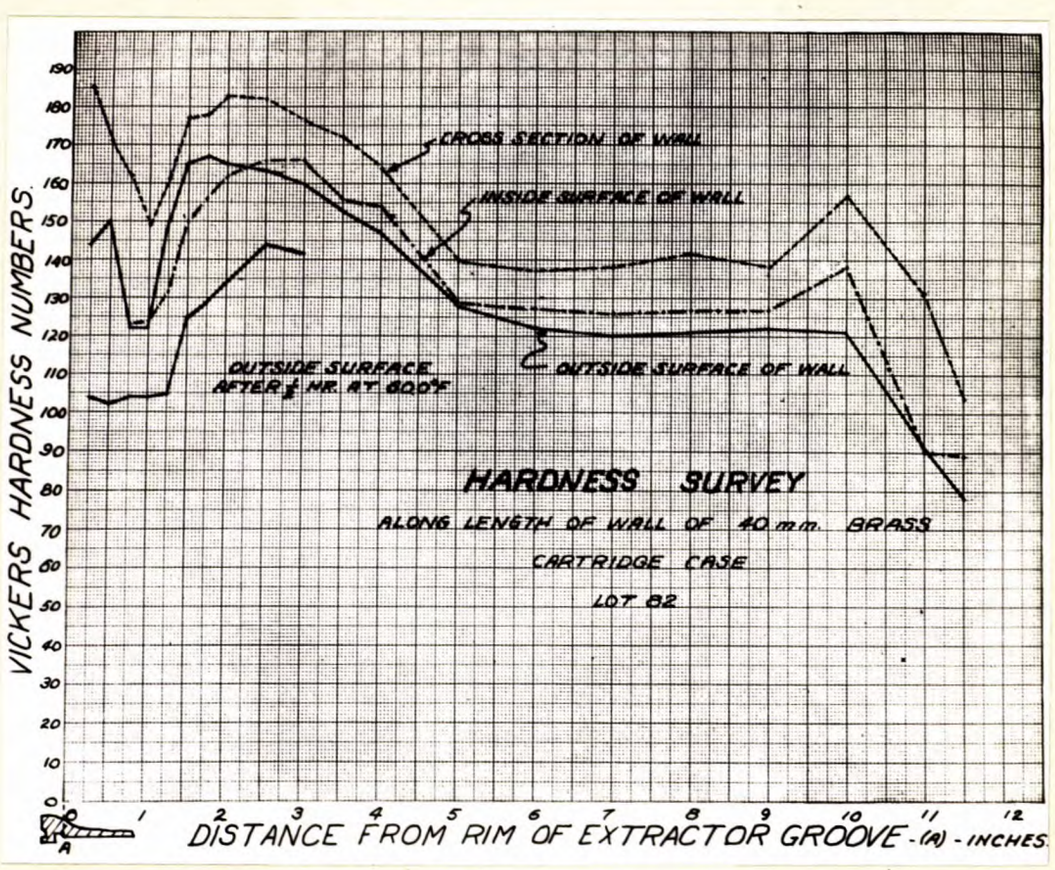
Figure 2.



VARIATION OF HARDNESS OVER LENGTH OF CASE, LOT NO. 82.
Note drop in hardness near base.

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Microscopic Examination:

Longitudinal sections, approximately ¼ inch wide, were cut from each case and prepared for microscopic examination. The samples were etched first in a chromium trioxide solution (2 grams CrO_3 + 2 cc. HCl + 200 cc. H_2O) and subsequently in ferric chloride solution (8 grams $FeCl_3$ + 25 cc. HCl + 100 cc. H_2O). Figures 3 to 8, photomicrographs at X75 magnification, show the structure at various locations of the longitudinal sections of the case from Lot No. 79. Since the structure of the section of the other case was similar, it is not shown. Figures 9, 10 and 11, photomicrographs at X250 magnification, show the structure near the base.

The microscopic examination reveals that the material near the base (Figures 4, 5, and especially 10) shows evidence of recrystallization. Farther away the material has a typical cold-worked structure (Figures 7 and 11). Figure 8 (near the mouth) shows annealed material.

(Figures 3 to 11 follow,
on Pages 6 and 7.
Text continues on Page 8.)

Figure 3.

Location, $\frac{1}{4}$ inch from base.

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Figure 4.

Location, $\frac{3}{4}$ inch from base.

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Figure 5.

Location, 1 inch from base.

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Figure 6.

Location, $1\frac{1}{2}$ inches from base.

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Figure 7.

Location, $2\frac{1}{2}$ inches from base.

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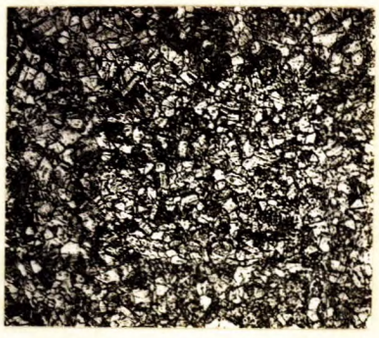
Figure 8.



Location, 8 inches from base.

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ABOVE PHOTOMICROGRAPHS ARE AT X75 MAGNIFICATION.

Etched in CrO_3 and $FeCl_3$ solutions.

Figure 9.

X250.

Location, ¼ inch from base.

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Figure 10.

X250.

Location, 1 inch from base.

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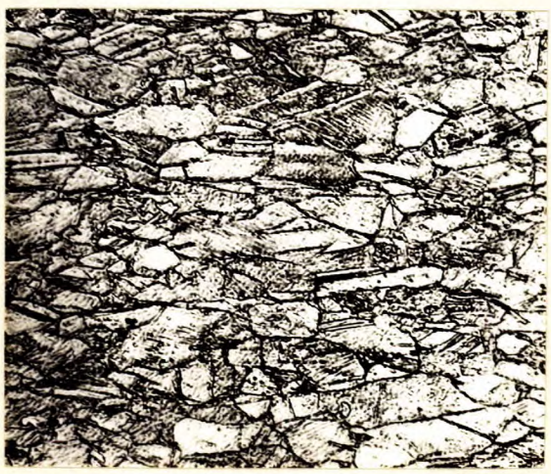
Figure 11.



X250.

Location, 1½ inches from base.

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ALL ABOVE ETCHED IN CrO_3 AND $FeCl_3$ SOLUTIONS.

X-Ray Diffraction:

The X-ray diffraction technique employing filtered copper radiation in conjunction with a back-reflection camera was used in an attempt to determine the cause of softening near the bases of these cartridge cases.

Patterns obtained from the cases from Lots Nos. 79 and 82 are given with detailed captions in Figures 12 to 15. In order to insure against spurious effects, some samples were examined in the unetched and macro-etched conditions and with filtered and unfiltered radiation.

Figure 12.

(CASE FROM LOT NO. 82).
Pattern obtained from a location one inch from the closest edge of the extracting groove.
Note the random dispersion of spots and the well-defined rings.

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Figure 13.

(CASE FROM LOT NO. 79).
Pattern obtained from a location one inch from the closest edge of the extracting groove.
Note well-defined rings.

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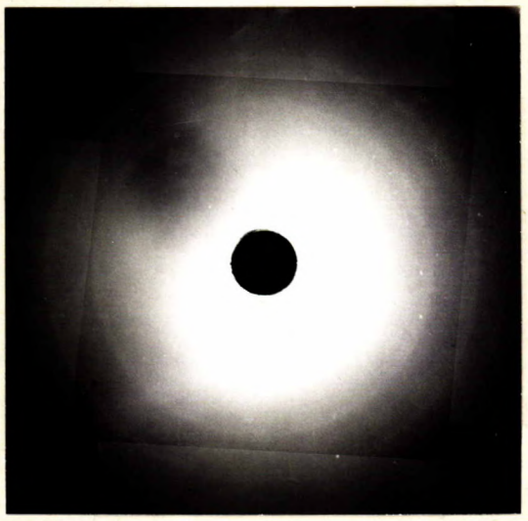
Figure 14.



(CASE FROM LOT NO. 79).

Pattern obtained from a location 2½ inches from the closest edge of the extracting groove.

Note diffuse rings.

The pattern from a similar location on the case from Lot 82 has the same appearance.

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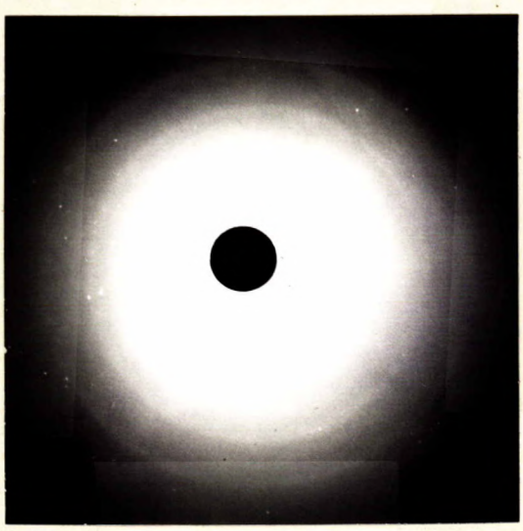
Figure 15.



(CASE FROM LOT NO. 79).

Pattern obtained from a location 2½ inches from the closest edge of the extracting groove.

This case was given a 570° F. draw for one-half hour. The drop in hardness at this location was 8 points Vickers. Note the presence of a few spots. Compare with Figure 14.

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Discussion:

The hardness of the material in the cases is higher in the centre of the cross-section of the wall than on either the inside or the outside surface of the wall (see Figures 1 and 2). This would indicate a variation in the degree of cold work received by the material, it being worked more in the centre than at either surface.

Both cases exhibit a marked drop in hardness at a point about one inch from the base. This may be clearly seen in Figures 1 and 2. This region near the base is cold-worked by two processes: the forging (heading) of the base, and the drawing of the walls. Thus, if the effects of these two processes overlap, or, on the other hand, do not meet, the region will receive either too much cold work or not enough. If it receives too much there is an excellent possibility that some recrystallization may occur during the stress-relief anneal, with a resultant softening of the material near the base. If it does not receive enough cold work, the material will again be soft.*

It may then be said that there are three possible explanations for the soft ring of material near the base of the cases: (a) the material received too much cold work and was in such a stressed condition that it recrystallized during the stress-relief anneal; (b) the stress-relief annealing temperature was too high, or the time at temperature too long; or (c) the material received insufficient cold work in this region and hence never attained the required hardness.

Microscopic examination showed some evidence of recrystallization during stress-relief annealing. The X-ray diffraction patterns obtained from these cases indicate also that

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* For a further discussion of this point, see article by D. H. Lloyd: "Some Principles of Cold-Working," in METAL INDUSTRY, London, Vol. 64, No. 1 (January 7th, 1944), pp. 2-5.

(Discussion, cont'd) -

recrystallization, rather than insufficient cold work, was the cause of the softening near the bases. This conclusion is based on the following observations:

The pattern (Figure 12) one inch from the extracting groove of the case from Lot No. 82 shows rather well-defined Debye-Scherrer rings with a background having individual interference spots. According to G. L. Clark, in "Applied X-Rays" (page 505; McGraw-Hill Book Co. Inc., 1940), these background spots appear when the grain size increases to around 10^{-3} cm., or larger, in diameter. The presence and perfection of these spots, then, indicate that there are rather large ordered arrangements of sub-grains in this part of the cartridge case. (By an ordered sub-grain structure is meant a rearrangement of the fragmented crystallites which has not proceeded far enough to be detected by ordinary microscopic practice.)

The possibility that in this case these interference spots were caused by insufficient cold work to fragment the grains rather than by the onset of recrystallization after fragmentation is ruled out by the microscopic appearance of locations one inch and 2½ inches from the base of the case from Lot No. 82 (Figures 5 and 7 respectively, and corresponding diffraction patterns Figures 12 and 14). It may be noticed that the photomicrograph at 2½ inches does not show as much directionality as the one at one inch, yet, in the diffraction patterns, the one-inch location shows a larger and more ordered sub-grain structure than the 2½-inch. When, as a check a section of a case from Lot No. 79 was drawn at 570° F. for one half hour, a drop in hardness resulted and some spots appeared at the 2½-inch location (see Figure 15). A pattern from the case drawn for one hour at 800° F. showed a random distribution of spots and rather sharp, discontinuous Debye-Scherrer rings. Also, it is commonly known that brass cold-worked to the extent

(Discussion, cont'd) -

of this one-inch location and not recrystallized will show continuous Debye-Scherrer rings because of fragmentation of the grains.

Since the pattern one inch from the extracting groove of the case from Lot No. 79 does not show background spots, the cold work, draw temperature, or grain size for this case was not the same as for Lot No. 82. This is in agreement with the fact that the hardnesses differ. The amount of cold work imparted and the subsequent annealing temperature largely determine some of the metallurgical characteristics of the sample and, consequently, the X-ray diffraction pattern obtained from it.*

CONCLUSION:

The great drop in hardness near the base of these cartridge cases was caused by recrystallization.

It is likely that the material in this region received an excessive amount of cold work and so was able to recrystallize during the normal stress-relief anneal. Another explanation might be that the stress-relief temperature was too high, or the stress-relief time too long. This point could not be checked due to lack of normal (good) cartridge cases.

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JPO:LPT:GHB.

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* For a discussion of a somewhat similar problem, see "The Estimation of Cold Work from X-Ray Diffraction Patterns," by Leopold Frommer, in The Journal of the Institute of Metals, Vol. 64 (1939), pp. 285-298.