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TECHNICAL NOTE

MRL-TN-411

EFFECT OF AGEING ON THE HARDNESS AND TENSILE PROPERTIES OF ALUMINIUM ALLOY 7001 WITH SPECIAL REFERENCE TO THE INFLUENCE OF DELAY TIME BETWEEN THE QUENCHING AND AGEING TREATMENTS

J.H. Cole

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MARCH, 197

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ABSTRACT

The change in hardness of aluminium alloy 7001 with time at temperatures 22°C, 50°C and 121°C following quenching from solution heat treatment at 460°C has been determined. At 22°C, natural ageing caused a hardness increase from 104 HV to 191 HV in 6½ years and the hardness was still rising. Ageing at 50°C caused an increase in hardness from 104 HV to 179 HV in 96 h and the hardness was still rising. After approximately 72 h at 121°C a peak hardness of 216 HV was reached. Subsequent heating at 121°C after various periods of time up to 4 years at 22°C and 96 h at 50°C caused an initial reversion in hardness.

Delay times varying from zero to 217 days at 22°C between quenching and ageing at 121°C for 24 h did not appear to have any significant effect on the hardness and tensile properties.

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POSTAL ADDRESS: Chief Superintendent, Materials Research Laboratories
P.O. Box 50, Ascot Vale, Victoria 3032, Australia

DOCUMENT CONTROL DATA SHEET

Security classification of this page: UNCLASSIFIED

1. *DOCUMENT NUMBERS:*	2. *SECURITY CLASSIFICATION:*
a. *AR Number:* AR-000-889	a. *Complete document:* UNCLASSIFIED
b. *Series & Number:* TECHNICAL NOTE	b. *Title in isolation:* UNCLASSIFIED
c. *Report Number:* MRL-TN-411	c. *Abstract in isolation:* UNCLASSIFIED

3. *TITLE:*
EFFECT OF AGEING ON THE HARDNESS AND TENSILE PROPERTIES OF ALUMINIUM ALLOY 7001 WITH SPECIAL REFERENCE TO THE INFLUENCE OF DELAY TIME BETWEEN THE QUENCHING AND AGEING TREATMENTS

4. *PERSONAL AUTHOR(S):* COLE, J.H.	5. *DOCUMENT DATE:* MARCH, 1978
	6. *TYPE OF REPORT & PERIOD COVERED:*
7. *CORPORATE AUTHOR(S):* Materials Research Laboratories	8. *REFERENCE NUMBERS:*
	a. *Task:*
	b. *Sponsoring Agency:* Munitions Supply Division
	9. *COST CODE:* 584770
10. *IMPRINT (Publishing establishment)* Materials Research Laboratories, P.O. Box 50, Ascot Vale, Vic.3039 MARCH, 1978	11. *COMPUTER PROGRAMME(S):* *(Title(s) and language(s)):*

12. *RELEASE LIMITATIONS (of the document):*

Approved for Public Release

12-0. *OVERSEAS:* N.O. [ ] P.R. [1] A [ ] B [ ] C [ ] D [ ] E [ ]

13. *ANNOUNCEMENT LIMITATIONS (of the information on this page):*

No Limitation

14. *DESCRIPTORS:*
Aluminium Zinc Magnesium Copper Alloy; Aluminium Alloys; Precipitation Hardening; Hardness; Tensile Properties.

15. *COSATI CODES:* 1106, 1308, 2012

16. *ABSTRACT (if this is security classified, the announcement of this report will be similarly classified):*

The change in hardness of aluminium alloy 7001 with time at temperatures 22°C, 50°C and 121°C following quenching from solution heat treatment at 460°C has been determined. At 22°C, natural ageing caused a hardness increase from 104 HV to 191 HV in 6½ years and the hardness was still rising. Ageing at 50°C caused an increase in hardness from 104 HV to 179 HV in 96 h and the hardness was still rising. After approximately 72 h at 121°C a peak hardness of 216 HV was reached. Subsequent heating at 121°C after various periods of time up to 4 years at 22°C and 96 h at 50°C caused an initial reversion in hardness.

Delay times varying from zero to 217 days at 22°C between quenching and ageing at 121°C for 24 h did not appear to have any significant effect on the hardness and tensile properties.

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EFFECT OF AGEING ON THE HARDNESS AND TENSILE PROPERTIES OF ALUMINIUM ALLOY 7001 WITH SPECIAL REFERENCE TO THE INFLUENCE OF DELAY TIME BETWEEN THE QUENCHING AND AGEING TREATMENTS

INTRODUCTION

Rocket motor bodies and closures for Rocket HEAT 66 mm L1A2 are being made in Australia from American Aluminum Association Alloy 7001 using production methods based on information supplied by A/S Raufoss Ammunisjonsfabrikker (RA). RA Inspection Plans Nos. 3-1332 and 3-1333 specify the following heat treatment: solution heat treat at 465 $^{+\ 5°C}_{-\ 10°C}$ for 30-40 min, quench into water at less than 40°C with a cooling time of 3-5 min, age at 120 ± 5°C for a minimum of 22 h with the delay time between solution heat treatment and ageing being either less than 2 h or greater than 48 h.

No published information on the effects of delay time on the properties of this alloy could be located. However, the recommendation is apparently based on reports that certain alloys of aluminium with zinc, magnesium and copper which contain chromium show changes in tensile strength and proof stress with variations of delay time between quenching and elevated-temperature ageing, particularly in the period 2 to 48 h (1,2).

To gain requisite information on the alloy and to investigate how critical the delay period was for this alloy, experiments were carried out to study (a) the change in hardness on ageing for various times at room temperature (22°C) and 121°C, (b) the change in hardness with delay time at room temperature followed by ageing at 121°C for various times, and (c) the variation in tensile properties with delay time at room temperature followed by a standard ageing treatment of 24 h at 121°C.

As motor body forgings may be held in hot (50°C) lubricant between solution heat treatment and the straightening and expanding operation, the variation in hardness with time at 50°C, and subsequent ageing at 22°C, was also studied. During the course of the above studies it was noted that the hardness of test pieces held at 22°C after short periods of ageing at 121°C, continued to rise. To determine whether the prior ageing at 121°C had changed the hardening mechanism, the effect of further heating at 121°C was also studied.

MATERIAL

The material used originated from Harvey Aluminum Co., USA, and was in the form of extruded bar 47.6 mm (1-7/8 in) in diameter; the composition is given in Table 1.

As there was a variation in macrostructure in the vicinity of the outer surface of the extruded bar, tensile test pieces were machined from 9.5 mm (3/8 in) diameter bars extracted from the central 35 mm (1-3/8 in) diameter region of the extruded bar. For comparison, a small number of test pieces containing some of the outer region of the bar which exhibited a coarse grain size after heat treatment were also tested. Discs, 3 mm (1/8 in) thick, were sliced from the extruded bar and cut into segments for hardness tests.

HEAT TREATMENT

The tensile blanks (9.5 mm (3/8 in) diameter bars) and hardness test pieces were heated in an air-recirculation furnace at 460°C for 40 min and quenched into water at 37°C; the water temperature rose to 39°C after 2 min. The samples were removed from the water and stored in an air-conditioned room at 22°C for room-temperature ageing. The ageing treatment at 50°C was carried out in an air-recirculation oven for various times from 1/4 h to 96 h. The ageing treatment at 121°C was carried out in the same air-recirculation oven for 24 h for the tensile blanks and for various times ranging from 1/4 h to 17 days for the hardness test pieces; for ageing times less than 1/4 h, an oil bath was used, the test pieces being quenched in white spirit after the ageing.

TESTING

The heat-treated hardness test pieces were abraded on abrasive papers to a 600 grade finish on surfaces normal to the extrusion direction. Vickers hardness tests were performed on these surfaces, away from the outer edge, using a standard diamond pyramid indentor and a load of 20 kg. The results of the hardness tests are given in Tables 2-4 and Figs. 1-5; individual values are the average of 3 readings, the variations being within ± 3 HV.

Standard proportional (WT1/16) test pieces 7.16 mm (0.282 in) nominal diameter with threaded ends were machined from the 9.5 mm (3/8 in) diameter bars after heat treatment; care was taken to avoid possible heating effects in the machining operations. The tensile tests were carried out on a Riehle universal testing machine using a cross-head speed of 1.3 mm/min (0.05 in/min), the results being given in Tables 5 and 6.

RESULTS AND DISCUSSION

(i) Hardness

The hardness in the as-quenched condition was 104 HV. Ageing at 22°C commenced with very little delay, as shown in Fig. 1; after 6½ years the hardness had reached a value of 191 HV and appeared to be still rising very slowly. On ageing at 50°C (Fig. 2), the hardness followed a pattern similar to that at 22°C but at a faster rate; after 96 h the hardness had reached 179 HV and was still rising. On standing at 22°C after ageing at 50°C (Table 2), the hardness continued to increase following the same hardness/time curve as if the hardness reached at 50°C had been reached at 22°C. On ageing at 121°C the hardness increased fairly rapidly, as shown in Fig. 3. The hardness reached a maximum of 216 HV after approximately 72 h and then fell to 202 HV after 17 days.

Test pieces held at 22°C for periods of 2 h to 1575 days had hardnesses ranging from 124 to 190 HV before being aged at 121°C (Table 3). On heating a sample held at room temperature for 2 h (HV 124) at 121°C the hardness remained virtually constant for 18 s and then increased to 135 HV after 72 s, the same hardness as a sample heated for 72 s immediately after quenching. On heating a sample held at room temperature for 48 h (155 HV) the hardness decreased to 142 HV after 72 s and then increased to 145 HV after 3 min, the same as a sample heated at 121°C for 3 min immediately after quenching. Other samples held for longer periods at room temperature behaved similarly (Table 3, Fig. 4). A test piece which had been aged for 95 h at 50°C (179 HV) and then held at room temperature for 554 days (191 HV) was also aged at 121°C. Again the hardness/time curve at 121°C followed a similar pattern, the hardness falling below the hardness reached at 50°C to a minimum value of 175 HV after 20 min and then increasing to 178 HV after 40 min, the same hardness as a sample heated for 40 min at 121°C immediately after quenching (Fig. 4). On further ageing at 121°C, the hardness/time curves for the above test pieces coincided with that for a test piece aged at 121°C immediately after quenching.

Test pieces aged for relatively short periods at 121°C increased in hardness on standing at room temperature and the hardness values were still rising slowly after 4 years (Table 4, Fig. 5). The increases in hardness were independent of the delay time between quenching and ageing at 121°C.

A test piece which had been aged for 15 min at 121°C (165 HV) and then held for 1200 days at 22°C (194 HV) was reheated to 121°C. The hardness fell to 182 HV after 20 min and then rose to 184 HV after 35 min, the same as that of a test piece aged for 50 min at 121°C immediately after quenching. Again, on further ageing at 121°C the hardness/time curve at 121°C coincided with that of a sample continuously aged at 121°C immediately after quenching (Fig. 4).

From the above results it may be inferred that hardening at 22°C and 50°C is caused by the formation of Guinier-Preston (GP) zones. These GP zones are dissolved causing a reversion in hardness by heating at 121°C, at which temperature precipitation takes place.

Although it would appear that material aged for the specified minimum time of 22 h at 121°C was close to maximum hardness it is slightly under-aged.

(ii) Tensile Properties

There was no trend in variation of tensile properties with time of delay between quenching and ageing at 121°C for 24 h. Test pieces taken from the centre of the extruded bar, which had a uniform fine grain size, exhibited typical uniform cup and cone fractures and the variations in properties were within the normal statistical scatter in material properties. The tensile properties of these test pieces, both in regard to average values and lowest recorded values, were well above the specified minimum values and the suggested typical values as shown in Table 5. The average elongation on a length of 4 x diameter was 9%, the lowest value being 8%, compared with the specified minimum value of 7%. On the other hand, test pieces taken from the outer regions of the extruded bar, which contained some coarse-grained regions, exhibited a less uniform fracture and longitudinal cracks were present in the coarse-grained regions of the fractures. Moreover, there was a scatter in the tensile properties, some values being just under the specified minimum values (Table 6).

The absence of any significant variation in tensile properties with variation of delay time between quenching and ageing at 121°C for 24 h is in accord with the hardness results which indicated that the atomic rearrangements responsible for the increase in hardness at 22°C were destroyed by heating at 121°C.

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TABLE 1

COMPOSITION OF MATERIAL, wt %

Element	Sample	Specification 7001
Copper	2.24	1.6 to 2.6
Iron	0.3 approx	0.40 max
Silicon	0.15 approx	0.35 max
Manganese	Faint trace	0.20 max
Magnesium	3.14	2.6 to 3.4
Chromium	0.2 approx	0.18 to 0.40
Zinc	7.41	6.8 to 8.0
Titanium	0.05 approx	0.20 max

TABLE 2

EFFECT OF AGEING TIME AT 50°C ON HARDNESS (HV 20), FOLLOWED BY AGEING AT ROOM TEMPERATURE (22°C)

Time at 50°C	Hardness, HV 20	Hardness (HV 20) after Various Times at 22°C, following Ageing at 50°C				
		4 h	24 h	48 h	7920 h	13 440 h
0	104	133	151	159	186	187
17 min	128	137	152	159	184	188
32 min	140	148	155	163	189	191
1 h	147	151	156	164	189	189
2 h	153	154	162	163	188	190
3 h	158				189	189
4 h	158		161	162	188	189
6 h	162				187	190
7 h	162				188	190
24 h	171		173		190	191
30 h	172					
48 h	173					
72 h	176					
96 h	179				190	190

TABLE 3

EFFECT OF AGEING TIME AT 121°C ON HARDNESS (HV 20)
FOLLOWING VARIOUS DELAY PERIODS AT 22°C
AFTER QUENCHING FROM 460°C

Delay Time, h	0	2	48	168	1032	5208	37800
Ageing Time at 121°C				Hardness			
Zero	104	124	155	164	176	183	190
10 s	107	123	154	163			
18 s	112	124	151	161			
36 s	124	127	143	156			
72 s	136	135	142	150			
108 s	138	138	142	145			
3 min	144	147	145	146			
4 min	149						
5 min							179
6 min	153		153	155			
10 min			159	159			171
15 min	164	164	165	166	165	165	173
30 min	175	173	175	175	175	175	174
1 h	186	186	182	183	181	181	184
4 h	202	203	201	199	203	200	
5 h		205					202
6 h						202	
7 h	209		208	204	205		
24 h	213	213	212	210	213	210	212
30 h	215	214					
48 h (2 days)	215	217			215	215	
72 h (3 days)	216	216		214			
96 h (4 days)			214				
120 h (5 days)					211	213	
144 h (6 days)	212	214	212		212		
168 h (7 days)					210		
192 h (8 days)	209	209	210				
240 h (10 days)	209	210		210			
360 h (15 days)			207				
408 h (17 days)	202	203					

TABLE 4

EFFECT OF AGEING AT ROOM TEMPERATURE (22°C) ON HARDNESS (HV 20) PRIOR TO AND FOLLOWING AGEING AT 121°C FOR VARIOUS TIMES

Ageing Time, h at 22°C	Hardness (HV 20)	Ageing Time, h at 121°C	Hardness (HV 20) After Various Times (h) at 22°C following Ageing at 121°C									
			0	24	72	240	1000	3700	9200	27600	33000	51000
0	-	0.25	165	167	168	171	177	187	190	195	193	200
2	124	0.25	164	164	167	168	175	184	189	195	193	199
48	156	0.25	165	166		170	178	185	190	194	192	196
168	163	0.25	169	168	169	173	178	186	191	195	194	195
1032	174	0.25	165			174		183	190	193	194	197
5232	181	0.25	165					187		195		198
37800	190	0.25	173									
0	-	0.5	176	176	175	176	180	185	190	196	198	199
2	124	0.5	173	176	177	177	180	186	192	197	196	199
48	156	0.5	175	176			178	187	189	195	198	196
168	163	0.5	175	175			182	188	191	198	195	196
1032	174	0.5	175					186	189	197	199	200
5232	181	0.5	175					191		200		202
37800	190	0.5	175									
0	-	1	186	185	185	185	186	188	192	197	199	202
2	124	1	186	186	186		187	190	193	199	198	205
48	156	1	182	182			185	186	192	195	195	198
168	163	1	183	184			186	187	193	198	196	199
1032	174	1	181			182		189	191	200	200	198
5232	181	1	181					191		198		200
37800	190	1	184									
0	-	4	202	201	203	203	201	202	203	205	207	204
2	124	4	203	204	203		203	204	203	206	203	208
48	156	4	201	203			203	203	204	205	205	20[illegible]
168	163	4	199	200			202	202	202	204	205	203
1032	174	4	203									
5232	181	4	200					205		205		207
37800	190	4	198									
0	-	7	209	205	207	206	208	206	206	210	210	210
2	124	5	205	206	204		206	205	207	209	211	209
48	156	7	208	207			207	207	209	210	208	208
168	163	7	204	205			207	209	205	209	209	208
1032	174	8	205									
5232	181	6	202									
37800	190	7	203									

TABLE 5

TENSILE PROPERTIES* OF MATERIAL FROM CENTRAL REGIONS OF BAR (UNIFORM FINE GRAIN SIZE) AFTER VARIOUS DELAY TIMES BETWEEN QUENCHING FROM 460°C AND AGEING AT 121°C FOR 24 h

Delay time, h	Limit of proportionality 1000 lbf/in^2	Proof stress 1000 lbf/in^2		Tensile strength 1000 lbf/in^2	Elongation per cent		Reduction of area per cent	Hardness HV 20
		0.1% offset	0.2% offset		on 4D	on 5.65 $\sqrt{A}$		
0.07	87	98.3	99.6	104.6	9	9	12	214
2	87	100.1	100.8	105.9	9	8		212
	87	99.6	100.3	105.1	9	9	12	212
4	88	99.6	100.8	105.7	10	9	13	214
8	74	97.2	98.3	104.0	10	9	12	211
	86	98.8	99.7	105.0	9	9	11	212
16	90	99.3	100.0	106.0	9	8	13	215
24 (1 day)	83	98.8	99.7	104.9	9	9	15	212
	87	98.3	99.7	104	10	9	12	215
	88	99.3	100	105	9	8	9	216
48 (2 days)	88	100.1	100.7	105.9	9	8	11	213
96 (4 days)	87	100.0	101	106.3	8	7	13	214
168 (7 days)	86	98.2	98.6	104.4	9	8	12	211
192 (8 days)	83	98.9	100	106	8	7	11	213
	91	99.8	101	106	9	9	14	214
1008 (42 days)	69	91.9	98.8	104	9	8	10	214
	82	98.7	99.3	105	8	8	10	213
5208 (217 days)	70	100.6	101	106	8	9	9	209
	69	98.8	99.7	105	10	9	14	210
Average value	83	99.2	100.1	105	9	8	12	213
Specified minimum values			82	89	7			
Suggested typical values (Ref. 3)			96.5	101	8			

* As the material conforms to an American Specification, the properties are expressed in non-SI units. For conversion, 1000 lbf/in^2 = 6.89 MPa.

TABLE 6

TENSILE PROPERTIES* OF MATERIAL FROM OUTER REGIONS OF BAR (NON-UNIFORM GRAIN SIZE) AFTER VARIOUS DELAY TIMES BETWEEN QUENCHING FROM 460°C AND AGEING AT 121°C FOR 24 h

Delay time, h	Limit of proportionality 1000 lbf/in^2	Proof stress 1000 lbf/in^2		Tensile strength 1000 lbf/in^2	Elongation per cent		Reduction of area per cent	Hardness HV 20
		0.1% offset	0.2% offset		on 4D	on 5.65 $\sqrt{A}$		
0.07	72	89.3	91.1	97.7	8	8	13	212
1.1	75	93	94.1	100	11	9	15	214
	66	78.2	81.8	90.8	9	8	18	211
4	65	76	79.8	89.1	13	12	-	213
16	75	93.1	94.1	99.8	10	9	12	213
	65	75.8	82.6	90.6	10	9	-	213
24	63	81.1	84.9	93.6	9	9	18	213
48	72	89.6	91.0	97.1	10	9	14	212
96	63	85.5	88.4	96.6	10	9	14	214
168	73	90.4	91.5	98.6	12	10	16	211
Average value	69	85.2	87.9	95.4	10	9	15	213
Specified minimum values			82	89	7			
Suggested typical values (Ref. 3)			96.5	101	8			

* As the material conforms to an American Specification, the properties are expressed in non-SI units. For conversion 1000 lbf/in^2 = 6.89 MPa.

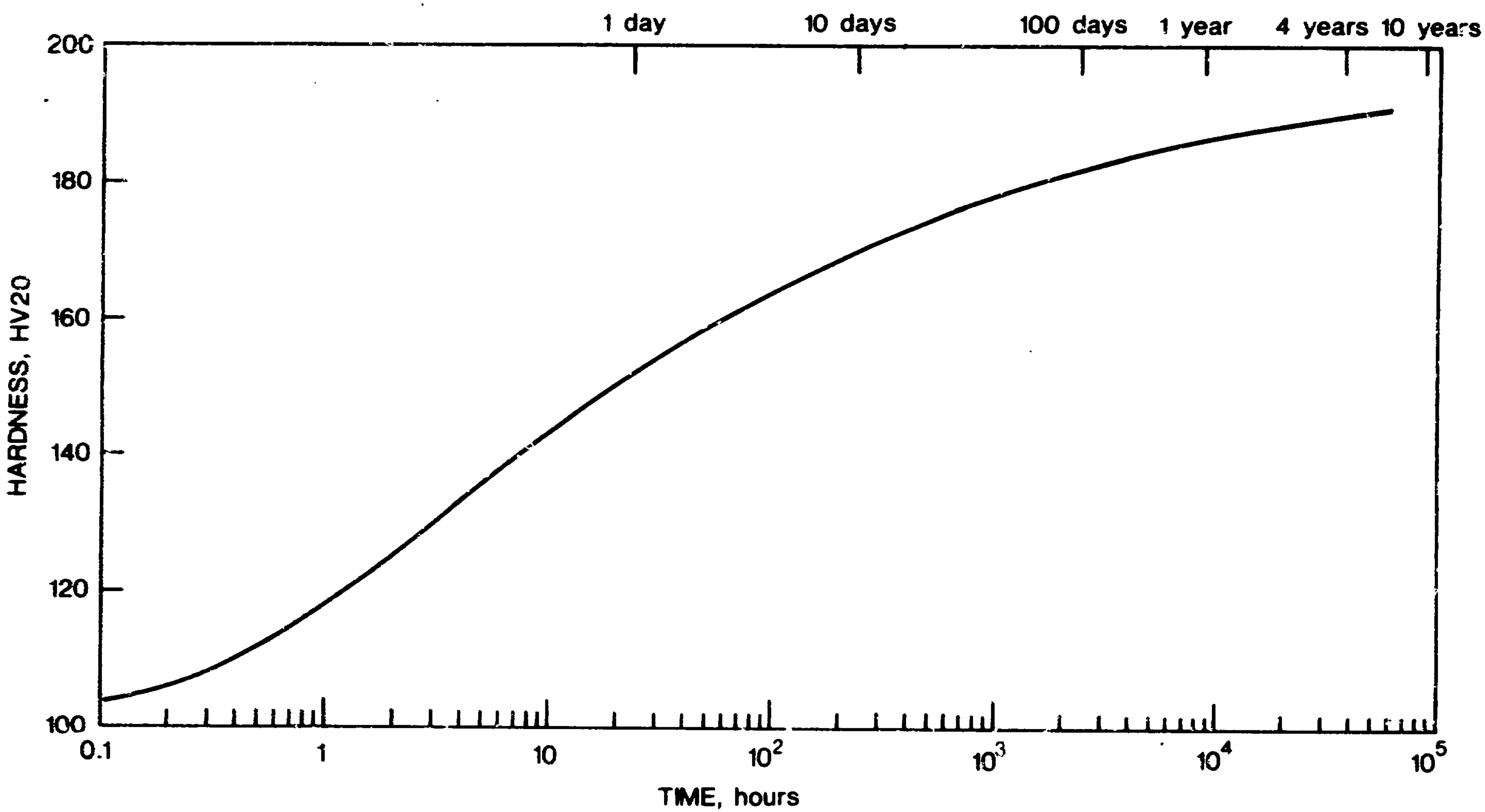

FIG. 1 - Changes in hardness with ageing time at 22°C.

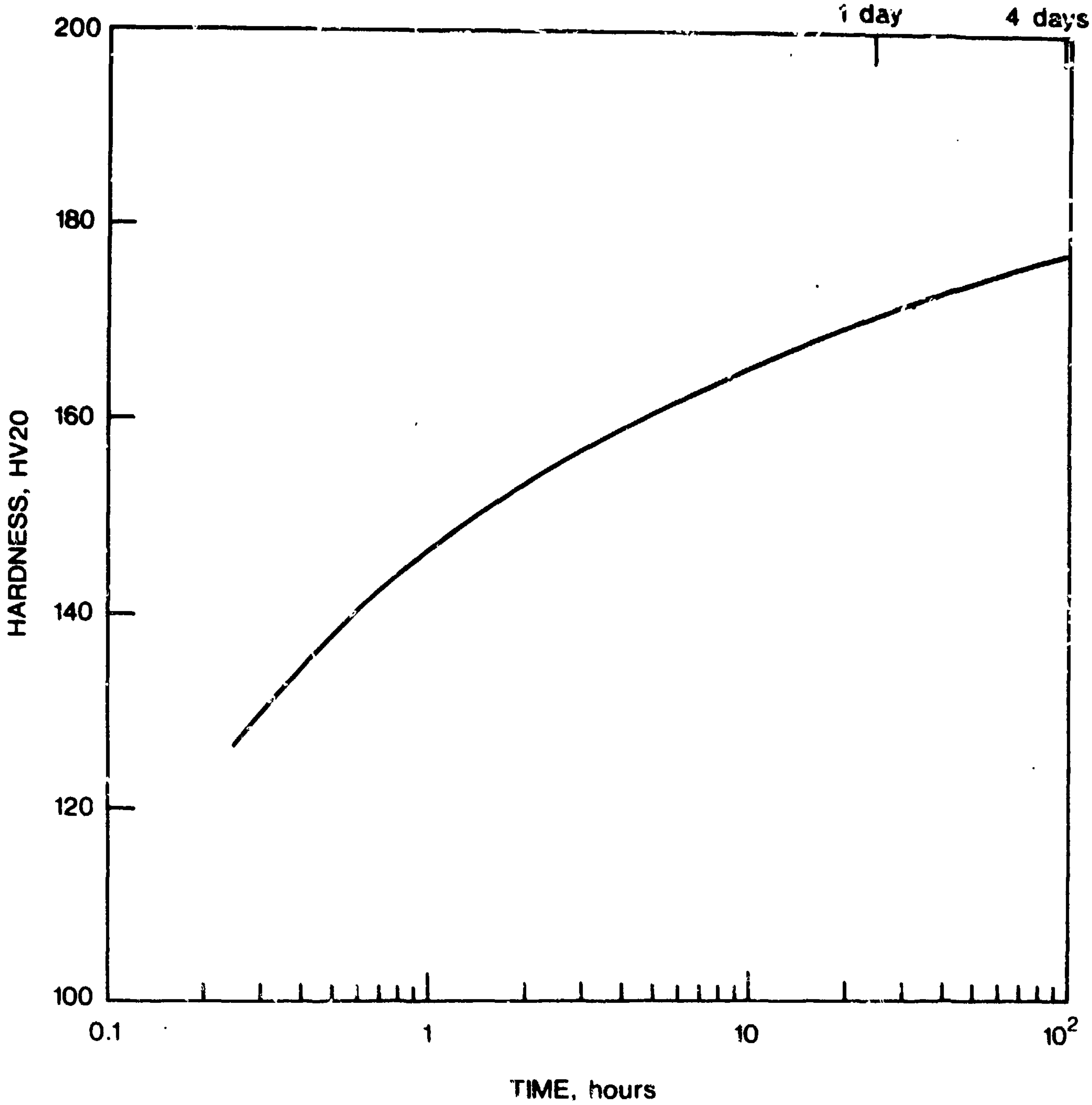

FIG. 2 - Changes in hardness with ageing time at 50°C.

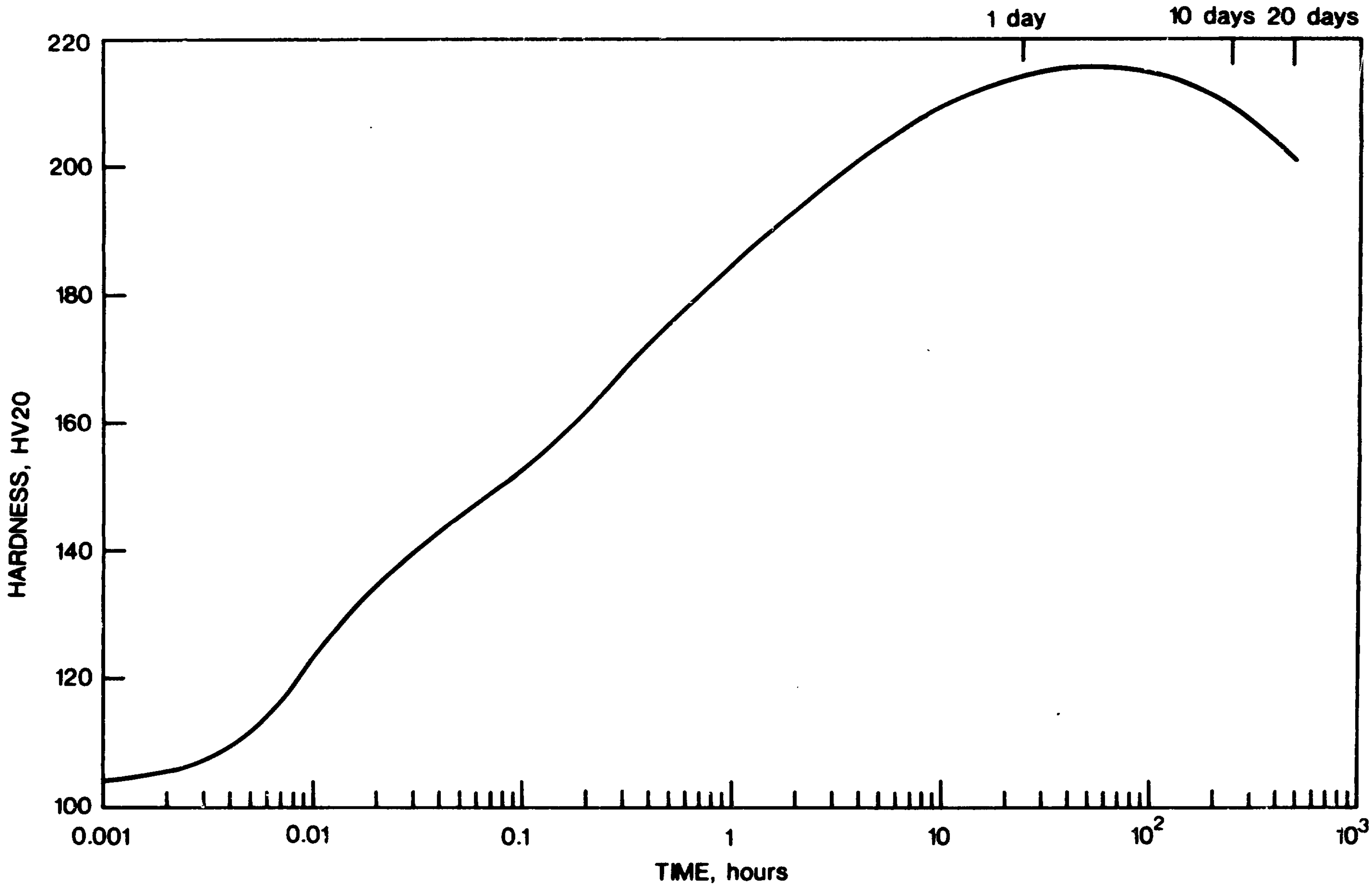

FIG. 3 - Changes in hardness with ageing time at 121°C.

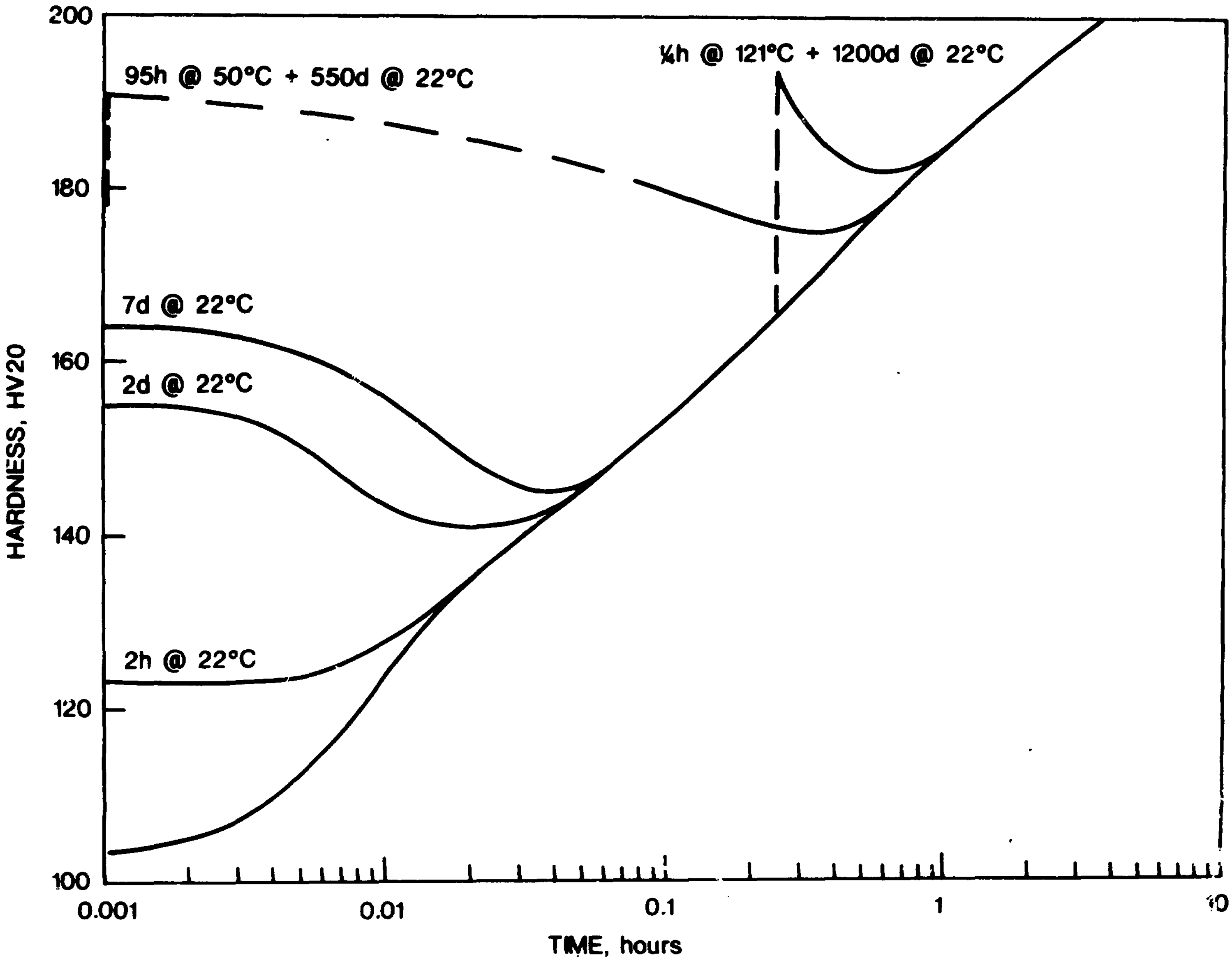

FIG. 4 - Changes in hardness with ageing time at 121°C after prior ageing at 22°C, 50°C, and 121°C.

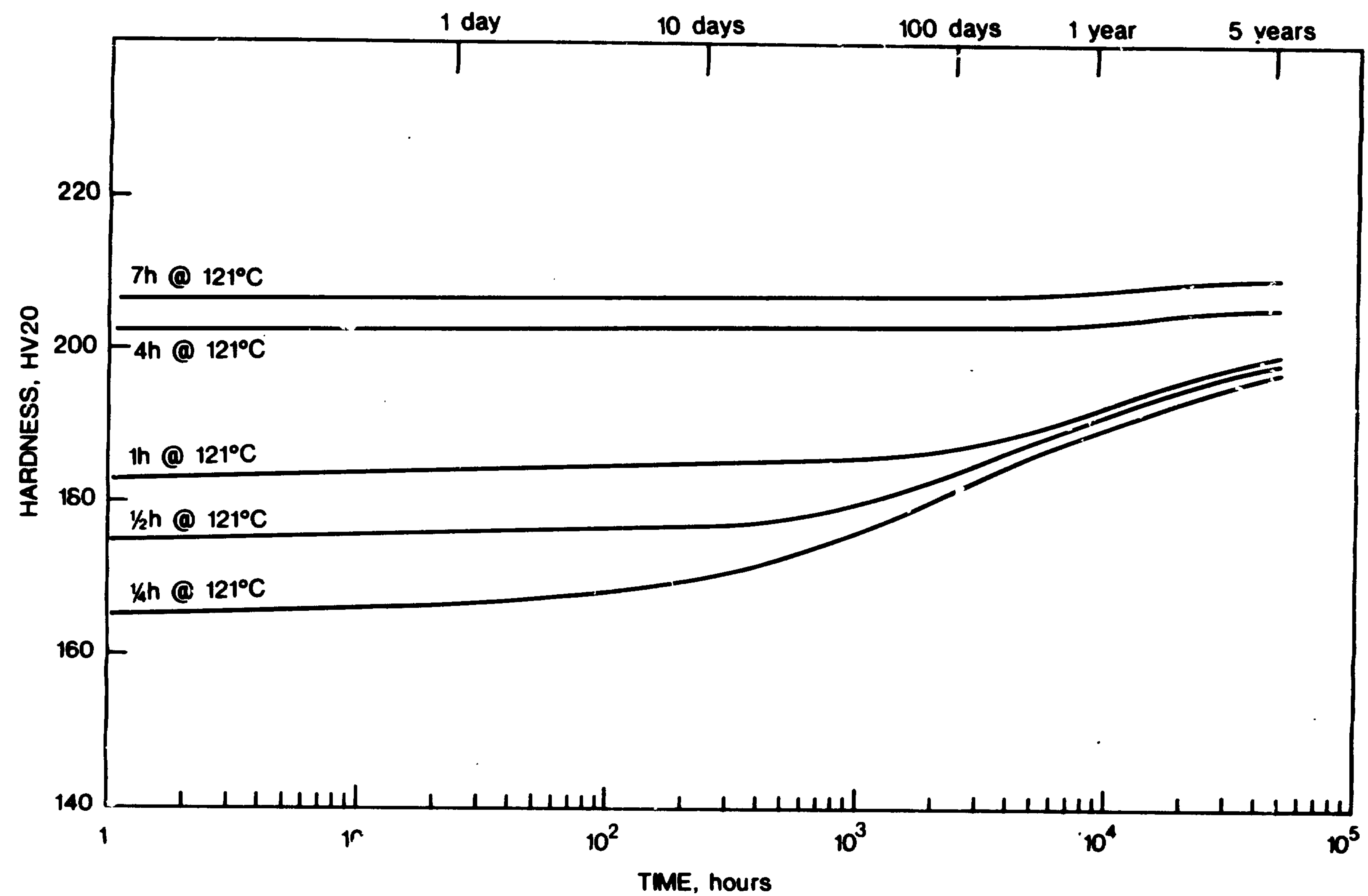

FIG. 5 - Changes in hardness with ageing time at 22°C after prior ageing at 121°C for various times.

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