

April 23, 1974

F. FERRIEUX

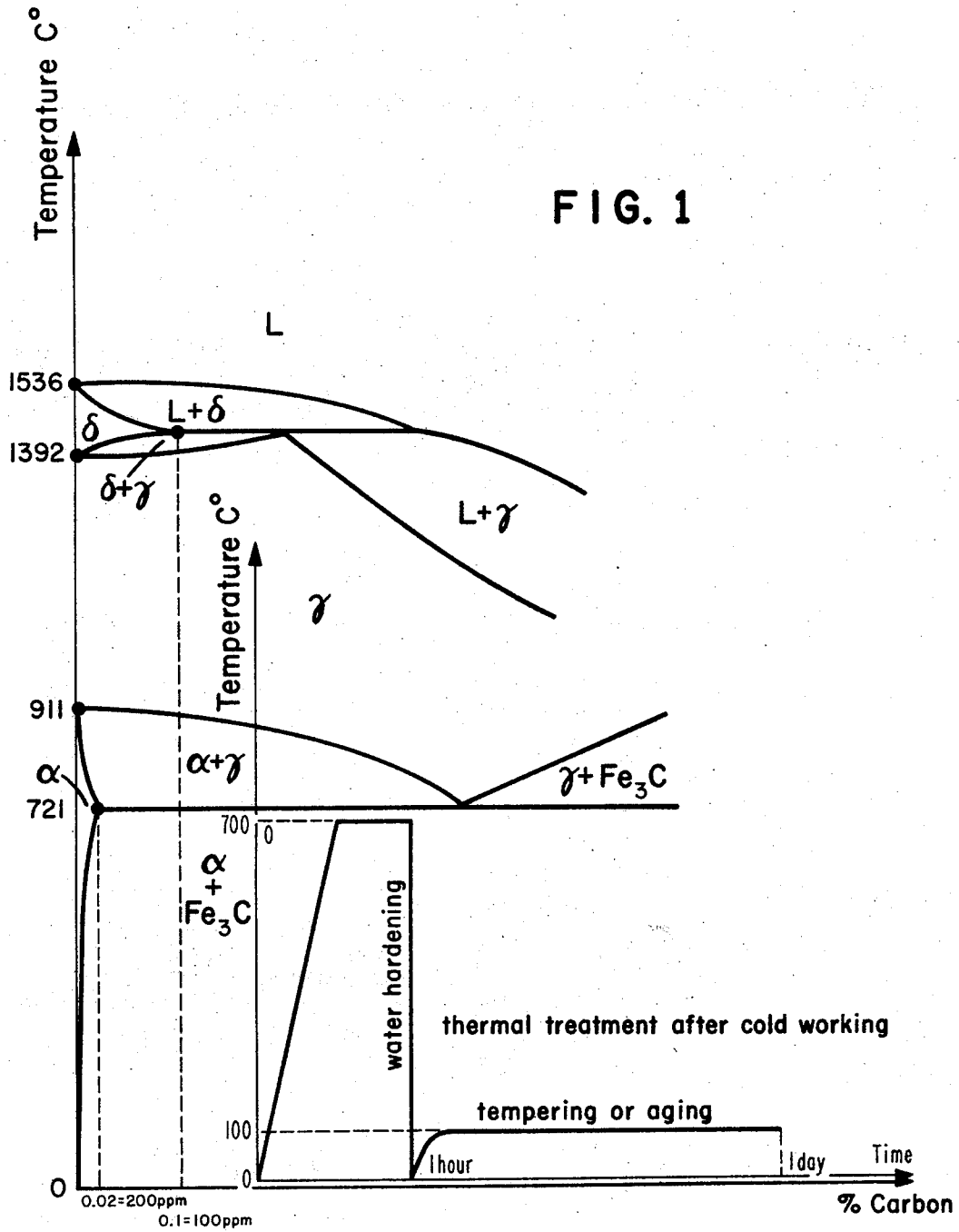
3,806,377

PROCESS ALLOWING COLD WORKING OF SOFT AND VERY SOFT  
OR LOW ALLOY CONTENT STEELS BY  
HARDENING THEM AFTER FORMING

Filed July 3, 1972

6 Sheets-Sheet 1

FIG. 1



April 23, 1974

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6 Sheets-Sheet 2

FIG. 2

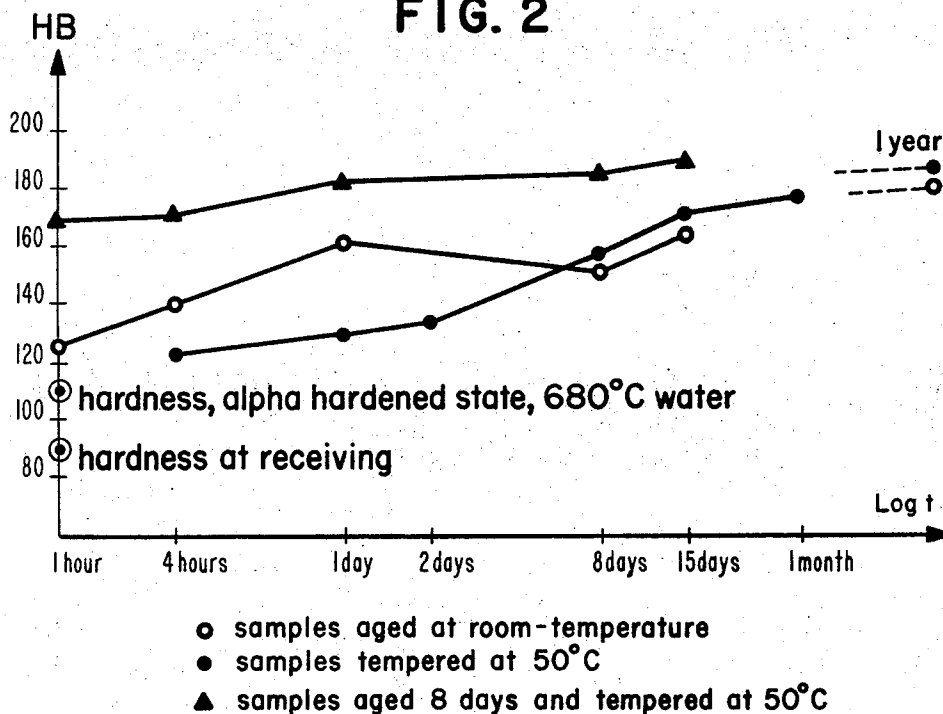
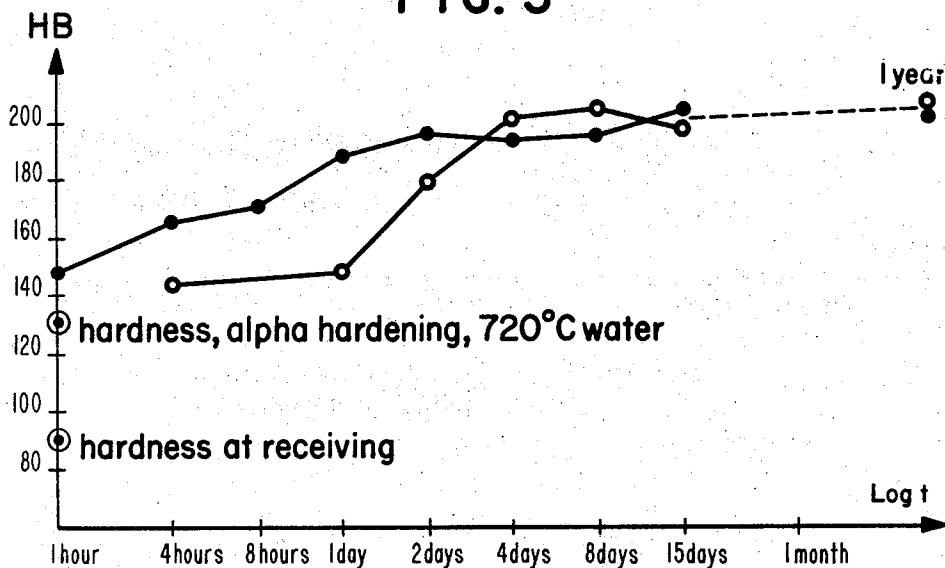


FIG. 3



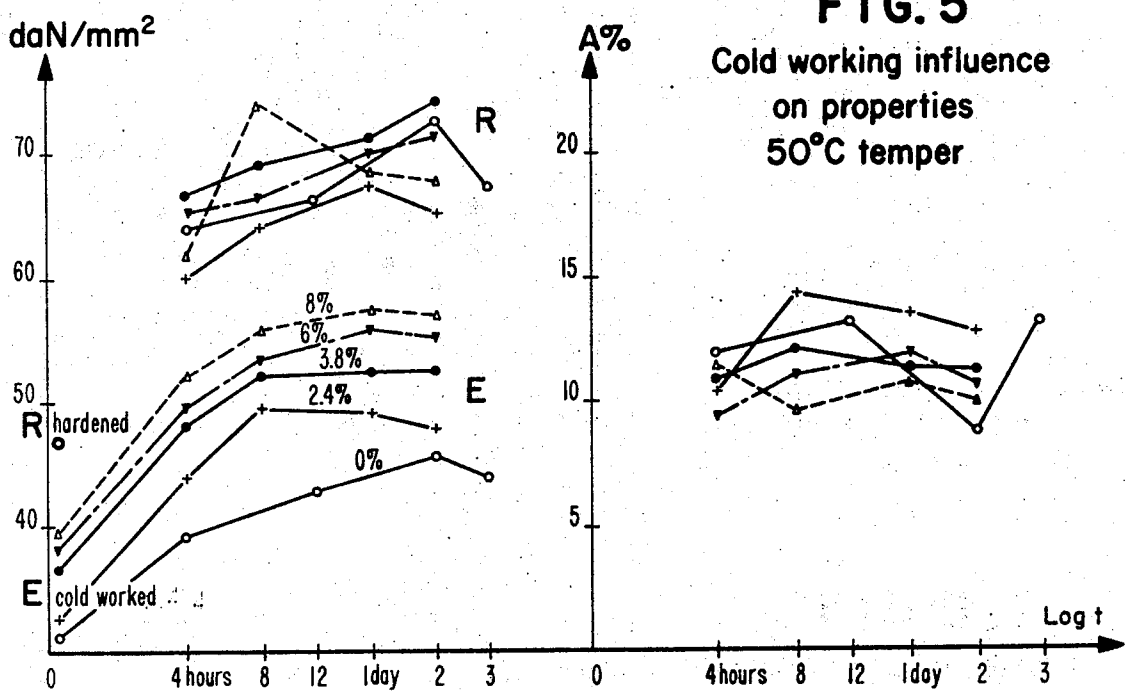
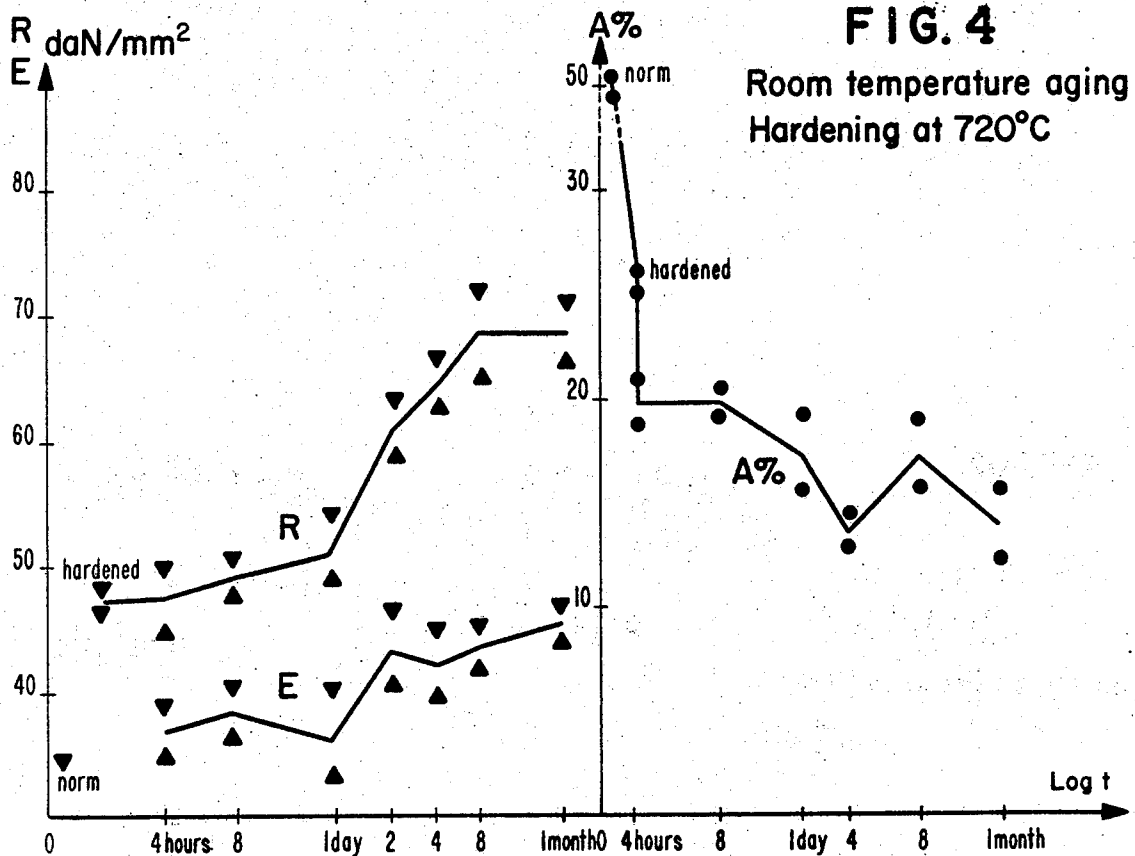
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6 Sheets-Sheet 3



April 23, 1974

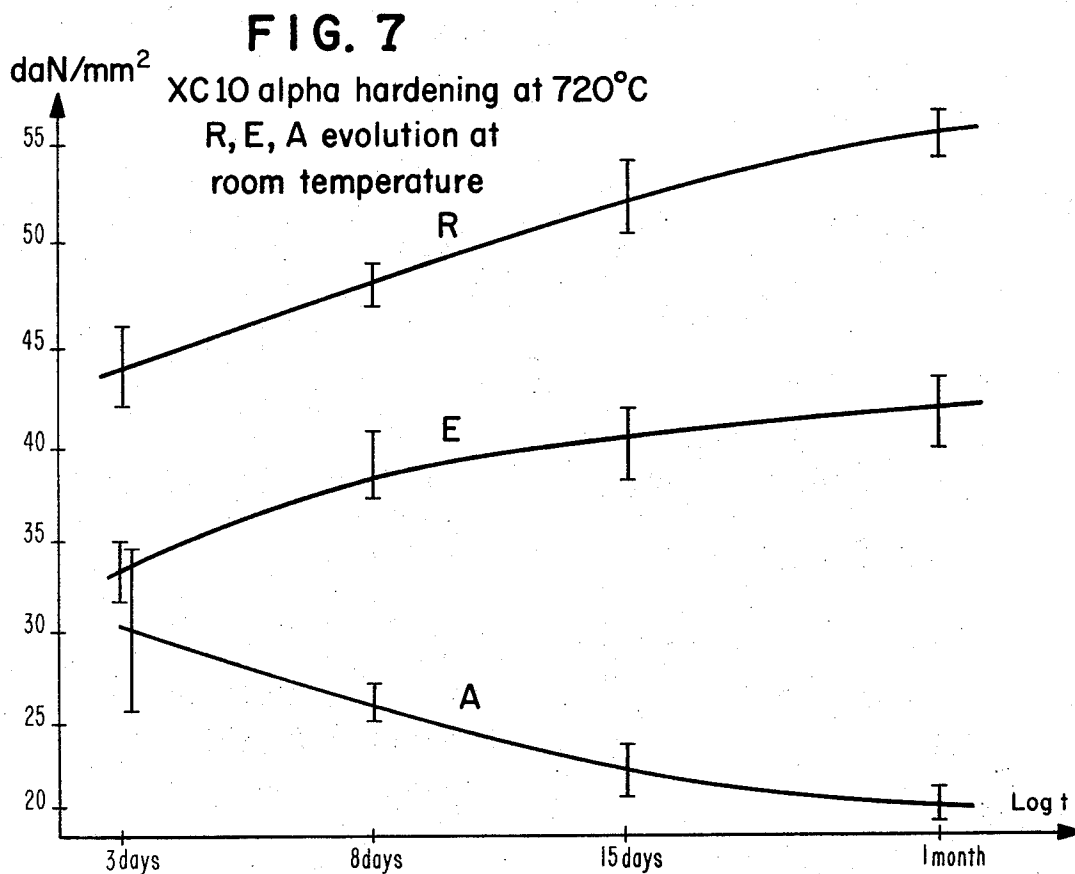
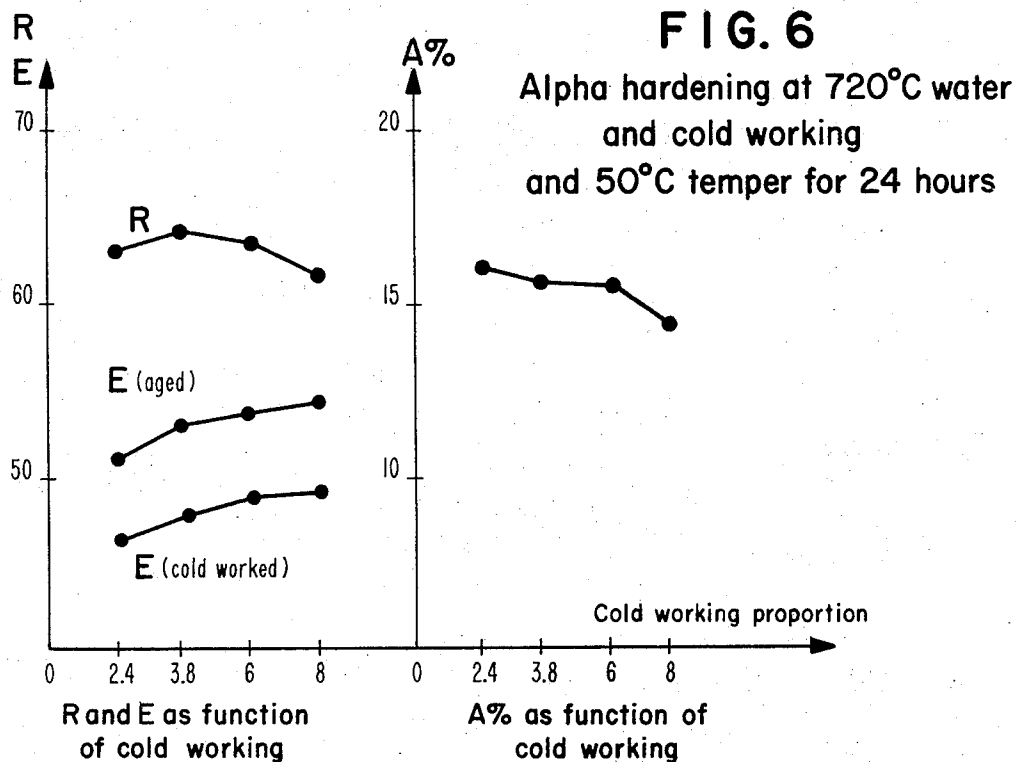
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Filed July 3, 1972

6 Sheets-Sheet 4



April 23, 1974

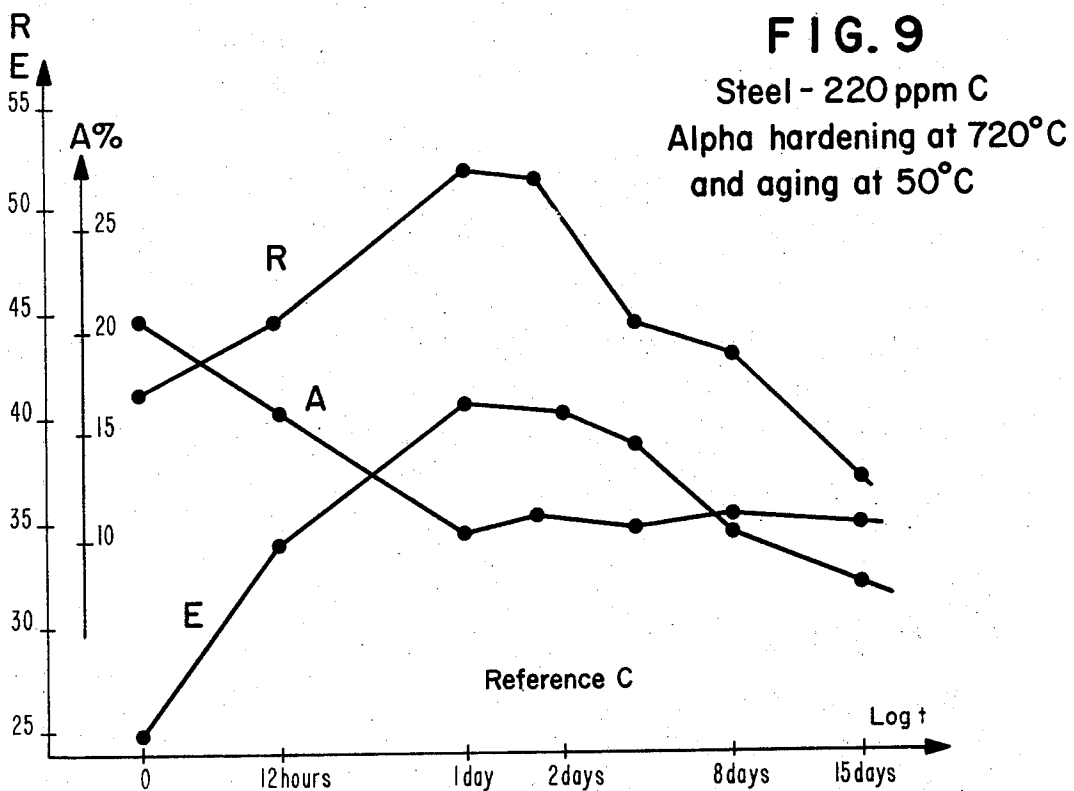
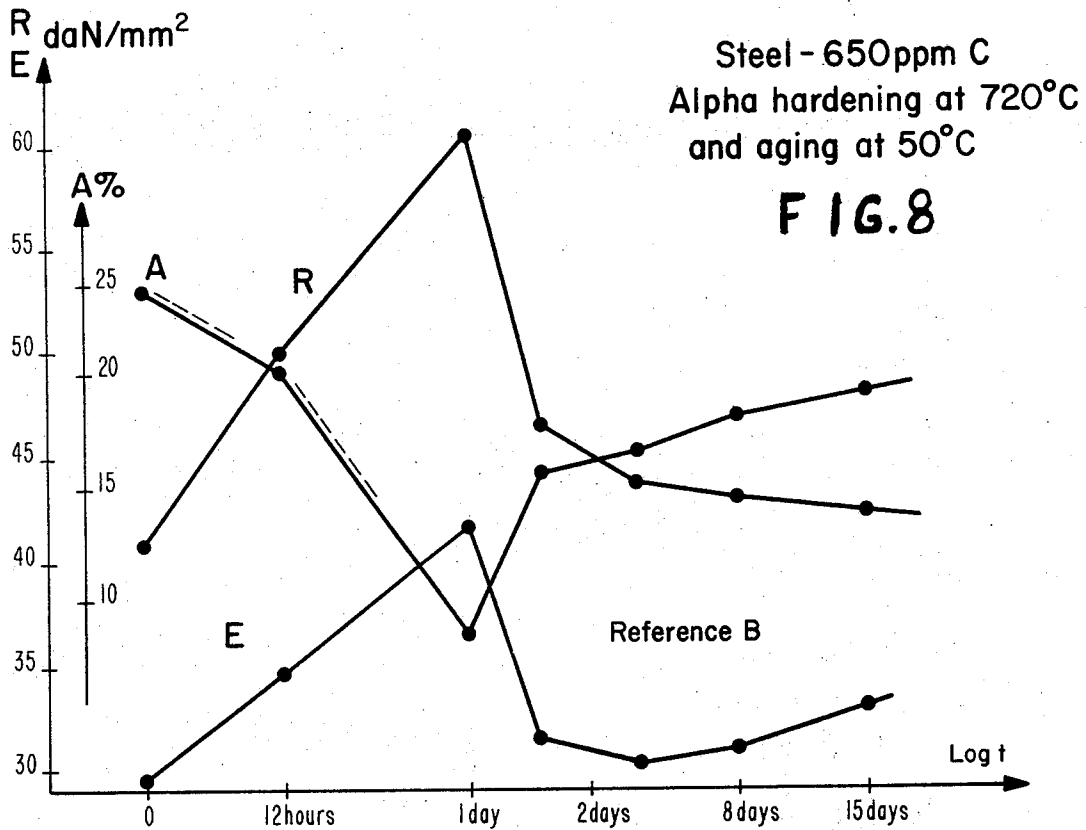
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Filed July 3, 1972

6 Sheets-Sheet 5



April 23, 1974

F. FERRIEUX

3,806,377

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Filed July 3, 1972.

6 Sheets-Sheet 6

TABLE 1 - XC 10 STEEL - Composition

| C         | SI       | MN        | P        | S        | AL        |
|-----------|----------|-----------|----------|----------|-----------|
| 0.06-0.11 | Max 0.10 | 0.20-0.40 | Max 0.03 | Max 0.03 | 0.03-0.08 |

TABLE 2 - VERY SOFT STEEL - Composition

|         | C     | MN   | SI    | AL   |
|---------|-------|------|-------|------|
| STEEL B | 0.065 | 0.30 | 0.016 | 0.03 |
| STEEL C | 0.022 | 0.25 | 0.011 | 0.03 |

1

3,806,377

## PROCESS ALLOWING COLD WORKING OF SOFT AND VERY SOFT OR LOW ALLOY CONTENT STEELS BY HARDENING THEM AFTER FORMING

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7124943

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16 Claims

### ABSTRACT OF THE DISCLOSURE

Parts are manufactured from soft and low carbon steel or low alloy steel by forming the part from the unhardened steel in its most malleable condition. Then the part is hardened and given a thermal aging treatment. The part may be aged at room temperature, it may be tempered at a temperature less than 70° C. or after aging at room temperature for one or two days it may be tempered at a higher temperature.

The present invention concerns the processes for obtaining given characteristics in metallic parts while minimizing forming or working endeavors. The aim of this invention is a combination of, one one hand, the forming or working of a part in the malleable state (annealed, standardized or restored), and on the other, achieving higher characteristics in finished parts or in parts yet to be finished. This is obtained by means of a thermal treatment involving aging after hardening, this aging being carefully placed within a gamut of fabrications and so allowing such combination.

There are three kinds of steel agings:

(1) Aging after hardening or rapid cooling: If a steel is heated to a temperature at which nitrogen solubility is significant such as above 400° C. or to a temperature at which the carbon solubility no longer is negligible such as above 600° C., and if the steel is rapidly cooled, nitrogen and carbon coalesce in the lattice and a supersaturated solid solution is obtained at ambient, which is harder than the saturated solid solution obtained by slow cooling. This supersaturated state lacks stability and causes progressive carbide and nitride precipitation if kept at ambient after hardening, or at a higher temperature. The carbides and nitrides will be the more minute the lower the temperature of the precipitation process. This fine precipitation causes gradual improvement, and a significant one, in the elastic limit, in the breaking-load and in hardness, with respect to the annealed state that is obtained when very slow cooling is employed. In the case of annealing the precipitates occur during cooling and most of them occur at high temperatures such as in excess of 400° C. and hence they are much coarser. The improvement in characteristics achieved by low temperature precipitation is caused by the fine size and homogeneous distribution of those precipitates. They cannot be shown under the optical microscope and may only be seen under an electronic one when precipitation is quite advanced as for example, several days at ambient or room temperature or one day at 50° C.

Thus the optical microscope will not allow one to distinguish between a steel that aged after hardening and a steel lacking hardening. If two steels of the same composition are considered, the one that did age will not or should not show in an optical manner the expected and computed amount of carbides.

With regard to the tempering temperature, there is a "transition" temperature (ca. 70° C.) above which the

2

hardening becomes less effective. There may, however, be applications for higher temperature temperings—whenever maximum strength is not the major concern since the resulting brittleness is much less. In any event, room temperature aging from one to two days before tempering at a temperature higher than that of the "transition" will provide excellent results. The moment the tempering temperature is higher than room temperature, one may achieve after some time a precipitate-coagulation leading to a lowering in the R, E, HB characteristics by means of a so-called "super-tempering."

(2) Aging after cold-working: If an annealed steel is cold-worked and if it is allowed to age at ambient, a gradual improvement in E, R, HB characteristics is observed as a function of time. Increasing the storage temperature only accelerates the phenomenon without affecting the end result—in contrast to aging after hardening. This phenomenon is slightly different from the one causing aging after hardening. Carbon and nitrogen still are the factors, but the latter predominantly so. Cold-working changes the thermodynamic equilibrium conditions of saturated solid solution  $\rightleftharpoons$  intermediate phases by causing inserted atoms to collect near the dislocations, and then a nitride precipitation mostly on those dislocations. Therefore, the "natural" aging is much less efficient than the one after hardening, as regards the mechanical properties.

Aside from this "natural" aging, which is due mostly to the nitrogen, an artificial aging may be achieved at a temperature between 100 and 300° C. In such case it is the carbon which causes the increase in the mechanical properties R, E, HB.

Steel users avoid the spontaneous and natural aging by making use of "killed" steels in which an element such as Al, Ti, Si, Zr, and the like stabilizes the nitrogen in the form of a nitride. Titanium also inhibits aging after hardening, provided its concentration is sufficient such as 0.5%.

(3) Aging after hardening and cold-working: This kind of aging may be considered a combination of the above two. The first stages of precipitation after hardening and cold-working much resemble those of aging after cold-working, while terminal precipitation resembles more closely aging after hardening. However the "transition" temperature in aging after hardening does not exist for aging after hardening and cold-working, and hence excellent characteristics may be obtained fairly rapidly at temperatures exceeding 70° C.

If the steel being used evidences Luder's level at cold working, cold-working must take place above that level.

Significant addition of titanium such as 0.5% inhibits aging after hardening and cold-working.

As regards the three kinds of aging above described, the active parameters do much differ from the structural hardening achieve at high temperatures in alloyed steels. In the latter case the hardening precipitates are of a complex nature based on a specific impurity; further, considering the high temperatures of these structural hardenings such as 400° C. to 600° C., the effects from a possible aging similar to one of the three kinds above mentioned are immediately eliminated.

To date, soft or very soft or low-alloy steels are endowed with those properties required for simple sold-working. This process carries with it a number of drawbacks, among which are the steel's brittleness, which limits the maximum achievable characteristics. Therefore, if solid qualities are required, fairly strong steels are necessary.

Spring steel manufacture is one application of aging after hardening of soft steels. Maximum tensile strength properties such as 90 kg./mm.<sup>2</sup> are required in a drawn

wire. Drawing takes place after hardening and maximum aging (tensile strength R: 60 kg./mm.<sup>2</sup>). With cold-working, one achieves R=85 kg./mm.<sup>2</sup>. Aging subsequent to cold-working allows gaining a few kg./mm.<sup>2</sup>. and therefore unkill steels are being used.

No use at all is made in such processes of the malleability properties of soft steels, since the deforming operation took place in a very hard phase of the material. What is being achieved here is the superposition in tensile strengths due to aging and due to cold-working.

The object of the present invention is to make use of the high malleability of the soft or very soft steels in the annealed state, then to endow these steels with the required mechanical properties after hardening, by means of a thermal aging treatment.

The process consists of:

(1) An alpha phase forming operation for the most malleable state desired (annealed, normalized or standardized, or simply restored).

(2) Dissolving carbon and nitrogen at temperatures exceeding 400° C. and more particularly falling within 600° C. and the point AC1 of the Iron-Carbon diagram, or else between this point AC1 and the initial melting point of the alloy. For a gamma phase solution, grain refining will be possible by making use of the transformation from alpha phase to gamma phase.

Dissolving of this nature also eliminates the cold-working effect of operation (1) if sufficient time is provided.

The time the carbon and nitrogen are kept in solution depends on the super-saturation being desired following hardening. This time no longer affects the mechanical properties after one hour has elapsed, except when dissolution took place below 500° C. or when recrystallization cannot be completed within one hour.

(3) Hardening in a fluid or by any process. The temperature and the fluid's nature depend on the desired rate of cooling, hence on the desired properties. Fast cooling will result in high tensile strength but low ductility after subsequent aging.

(4) A possible controlled cold-working higher than the Luder level, which might be a calibration stage. Tempering temperature should be less than 100° C., so as to avoid aging after cold-working as due to carbon remaining even in killed steels.

(5) Room temperature aging for a minimum time which depends on operating conditions (2) and (3). Room temperature evolution of properties slows down much after a month and becomes infinitesimal after three.

(6) Tempering: in lieu of room temperature aging, higher temperature tempering is possible. Evolution of properties evolves much more rapidly. The hardening obtained is the same if tempering temperature is below 70° C. In the opposite case, a softer but also less brittle product is obtained, except for operation (4) being carried out in its time.

(7) Room temperature aging for one or two days followed by tempering at a temperature exceeding 70° C. In this case aging causes a large number of nuclei to appear and tempering causes precipitation on those nuclei. This procedure achieves maximum increase in the elastic limit.

The process forming the object of the invention particularly applies to killed steels (for instance with aluminum though not with titanium, since aging after hardening would be decreased). Because natural aging is absent from these steels, difficulties during operation (1) for cold-working are avoided. The lower the carbon content, the more profitable the operation, the ductility being the higher while the mechanical properties remain the same.

The process forming the object of the invention may be applied to any manufacturing procedure comprising a cold-working operation of phase alpha (forging, swaging, fluo-lathing, etc.).

#### EXAMPLE 1

As a non-limiting illustration, we are showing the results obtained with a soft steel, type XC 10, the composition of which is provided in Table 1. This is a steel killed with aluminum.

FIG. 2 shows the evolution of hardness during room temperature aging and of 50° C., tempering following dissolution at 680° C., and water hardening.

FIG. 3 shows this evolution for a dissolving temperature of 720° C.

#### EXAMPLE 2

Illustratively shown are the properties R (tensile strength), E, A percent which were obtained after diverse alpha hardening treatments followed by room temperature and 50° C. agings, for the same steel as in Example 1. FIG. 4 shows the evolution of those properties at room temperature after dissolution at 720° C. and remaining at this temperature for half an hour, then water hardening. It will be observed that an elastic limit exceeding 40 kg./mm.<sup>2</sup> may be obtained without cold-working, for an elongation remaining larger than 10%. The small value of the rupture load in the standardized condition suggests the magnitude of the efforts required for cold-working prior to treatment. FIG. 5 shows the evolution of the same properties at 50° C. when 0-8% cold-working is added between hardening and tempering.

It will be noted that the hardening from cold-working adds to that from aging. The usefulness of such cold-working consists in allowing stabilizing the mechanical properties. The elastic limit remains at a reasonable value for the hardened state, and this cold-working does not require excessive shaping efforts whereas the final elastic limit after aging is very high.

FIG. 6 shows the effect of this cold-working upon the properties after standard annealing of 24 hours at 50° C.

#### EXAMPLE 3

The results shown in Examples 1 and 2 are valid for thicknesses of the order of those of our tensile test pieces of 3 mm. diameter. For larger thicknesses, core cooling rates may be considerably less and less useful mechanical properties may be expected. FIG. 7 shows one kind of bar cold-forged following annealing, then processed according to theory at 720° C. for half an hour and water-hardened, then tempered at 50° C. The tensile test piece walls were machined following hardening and aging. These parts were hollow cylinders and with 100 mm. diameters and 13 mm. thicknesses. When FIGS. 4 and 7 are compared, it will be observed that the elastic limit in the latter, which is obtained after one month at 50° C., is less by 3-4 kg./mm.<sup>2</sup> than the one obtained from faster cooling. A more significant drop is observed in the value of the rupture load, which decreased about 12 kg./mm.<sup>2</sup>. Be it noted that for elongations exceeding 20%, the elastic limit remains very high.

#### EXAMPLE 4

Besides the case of XC 10 steel, the most interesting results apply to very low-carbon steels which are easily cold-workable. Table 2 shows the composition of two very soft steels which were studied for their aging behavior.

FIG. 8 shows the evolution at 50° C. after alpha-hardening at 720° C. of reference steel B, which held the most carbon (0.06%).

The elastic limit of this steel in the annealed state is 17 kg./mm.<sup>2</sup>; elongation is 42% and the rupture load is 26 kg./mm.<sup>2</sup>. After one day at 50° C., the rupture load is 60 kg./mm.<sup>2</sup> and the elastic limit is 42 kg./mm.<sup>2</sup>, elongation now being only 8%.

FIG. 9 refers to a steel holding less carbon. After one day of aging at 50° C., the rupture load is less than in the preceding case, but the elastic limit is of the same order and the elongation exceeds 10%.

When these results are compared with those obtained from the XC 10 steel, it will be noted that the maximum elastic limit achieved for 50° C. annealing is 42 kg./mm.<sup>2</sup> in every case. This value therefore may be expected in the absence of any cold-working between hardening and tempering when soft or very soft, thin steel parts are being used whereas the elastic limit is three times less after annealing.

What is claimed is:

1. A process for manufacturing metallic parts of steel with low carbon content of less than 0.3% and with either a low-alloy content or none at all, whereby the manufacturing takes place as cold-working and subsequent thermal treatment that minimizes the required shaping efforts in order to give the final mechanical properties, this process comprising:

- (a) shaping said steel while in the malleable state in one or several stages with an intermediate annealing if necessary, whereby a shaped part is obtained,
- (b) heating said shaped part to a temperature exceeding 400° C. but less than the initial melting point of the alloy, whereby any carbon or nitrogen are dissolved in the steel,
- (c) hardening said heated part by cooling the part with a fluid, and
- (d) room temperature aging said part or tempering at a temperature between room temperature and 150 C.

2. A process according to claim 1 wherein the heating temperature is between 600° C. and the point AC1 of the iron-carbon diagram.

3. A process according to claim 1 wherein the dissolving occurs in the gamma or delta phase, that is, at a temperature between AC1 and the initial melting point.

4. A process according to claim 1 wherein the steel is killed with aluminum, silicon or zirconium but excluding titanium.

5. A process according to claim 2 wherein the steel has a very low carbon content of less than 0.06%.

6. A process according to claim 2 wherein the tempering takes place at a temperature less than 70° C.

7. A process according to claim 2 wherein the tempering is achieved in two steps with prior room temperature aging for 1 or 2 days followed by higher temperature tempering.

8. A process according to claim 2 wherein cold-working at a level higher than the Luder level is undertaken between hardening and tempering when a Luder level exists at that moment, this cold-working taking place within two days after hardening, and in which process the tempering temperature is less than 100° C.

9. A process according to claim 8 wherein cold working is caused by a calibration stage.

10. A steel product obtained by the process of claim 2.

11. A steel product obtained by the process of claim 4.

12. A steel product obtained by the process of claim 5.

13. A steel product obtained by the process of claim 6.

14. A steel product obtained by the process of claim 7.

15. A steel product obtained by the process of claim 8.

16. A steel product obtained by the process of claim 9.

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