

# Tempering Characteristics of a Vanadium Containing Dual Phase Steel

M. S. RASHID and B. V. N. RAO

Dual phase steels are characterized by a microstructure consisting of ferrite, martensite, retained austenite, and/or lower bainite. This microstructure can be altered by tempering with accompanying changes in mechanical properties. This paper examines such changes produced in a vanadium bearing dual phase steel upon tempering below 500 °C. The steel mechanical properties were minimally affected on tempering below 200 °C; however, a simultaneous reduction in uniform elongation and tensile strength occurred upon tempering above 400 °C. The large amount of retained austenite (~ 10 vol pct) observed in the as-received steel was found to be essentially stable to tempering below 300 °C. On tempering above 400 °C, most of the retained austenite decomposed to either upper bainite (at 400 °C) or a mixture of upper bainite and ferrite-carbide aggregate formed by an interphase precipitation mechanism (at 500 °C). In addition, tempering at 400 °C led to fine precipitation in the retained ferrite. The observed mechanical properties were correlated with these microstructural changes. It was concluded that the observed decrease in uniform elongation upon tempering above 400 °C is primarily the consequence of the decomposition of retained austenite and the resulting loss of transformation induced plasticity (TRIP) as a contributing mechanism to the strain hardening of the steel.

## I. INTRODUCTION

DUAL phase steels are characterized by a microstructure that consists of 75 to 85 vol pct fine grained ferrite with the balance being a uniformly distributed mixture of martensite, retained austenite, and lower bainite<sup>1-7</sup> in varying proportions. These steels are currently manufactured and used commercially. They are produced by continuously annealing certain low carbon Si-Mn<sup>4</sup> or microalloyed high strength, low alloy (HSLA) steels,<sup>1,2,3</sup> or directly off the hot mill.<sup>6</sup> The continuous annealing generally consists of heating the steel for a short time at temperatures in the ferrite-austenite, ( $\alpha + \gamma$ ), or austenite, ( $\gamma$ ) regions of the Fe-C phase diagram and cooling to room temperature. The cooling rate may be in the range between air cooling and water quenching depending on steel chemistry, available facilities, and desired mechanical properties.

The stress-strain behavior of dual phase steels<sup>2</sup> is characteristically different from that of ferrite-pearlite steels such as plain carbon steel or the HSLA steels (Figure 1). The ferrite-pearlite steels have yield point elongation, a high yield strength to ultimate tensile strength (YS/UTS) ratio, and their strength and ductility (uniform elongation) are inversely related (Figure 2). Dual phase steels have a continuous stress-strain curve with no yield point elongation. They work harden very rapidly at low strains, have a low YS, a high UTS, and hence a low YS/UTS ratio. They have better formability than the ferrite-pearlite steels of equivalent tensile strength, and their strength-ductility data fall on a separate curve (Figure 2) than that for ferrite-pearlite steels.

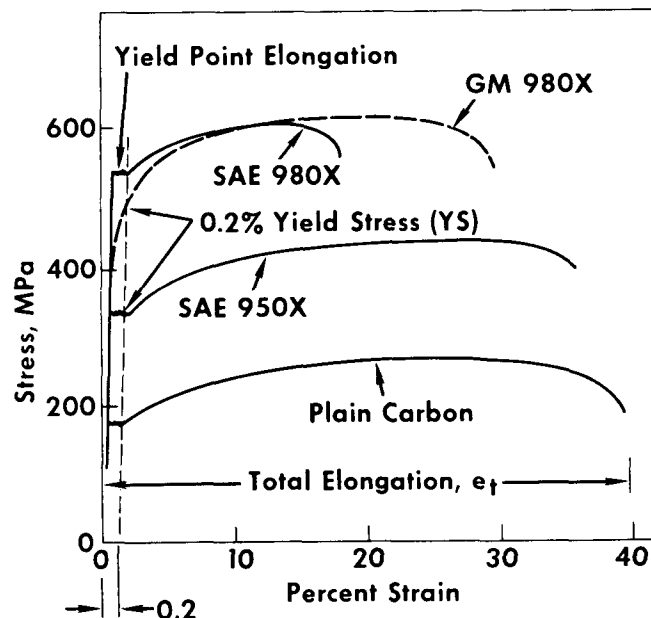


Fig. 1—Schematic stress-strain curves for plain carbon, HSLA, and dual phase steels. SAE 950X and 980X are Society of Automotive Engineers designations for HSLA steels of different strength levels. GM 980X is a General Motors developed dual phase steel. GM 980X is more ductile than SAE 980X although both steels have similar tensile strength.

The mechanical properties of dual phase steels can be altered by low temperature heating, or tempering which produces various microstructural changes in the steel. Tempering has been successfully used as a post-continuous annealing step to improve the strength-ductility relationship of certain microalloy-free dual phase steels.<sup>4</sup> Also, elevated temperatures might be experienced during service use of the steels, and knowledge of any mechanical property change resulting from such exposure is desirable. However, data are not publically available on the relationship between mechanical properties and tempering treatments as a function of steel composition and processing parameters, or on the

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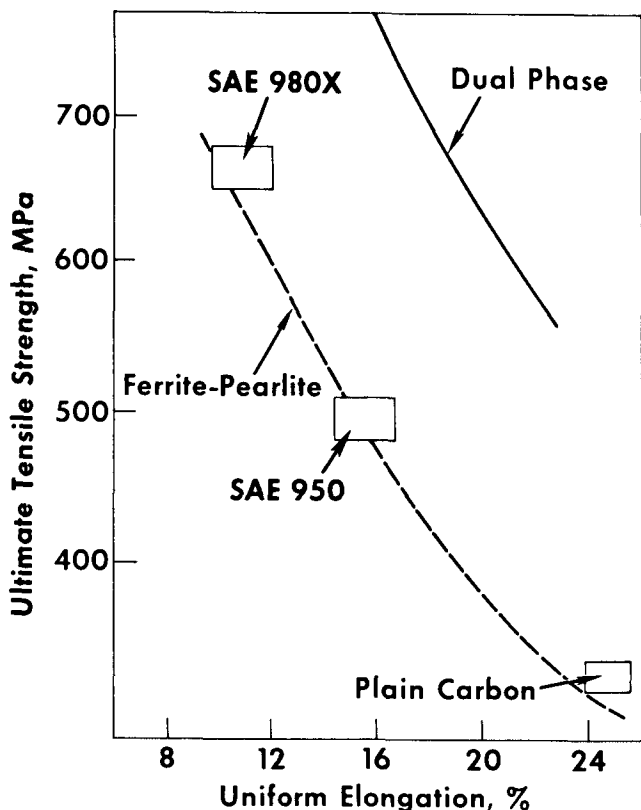


Fig. 2—Strength-ductility relationship of dual phase steels compared with that for plain carbon and HSLA steels. The dual phase steel curve is far above that for ferrite-pearlite steels (Ref. 5).

phase transformations and other microstructural changes that can be produced by tempering.

This paper examines the mechanical property changes produced in a dual phase steel by tempering below 500 °C to determine whether mechanical properties can be further improved. The material studied was a laboratory produced, vanadium bearing dual phase steel (0.1 pct C, 1.5 pct Mn, 0.5 pct Si, 0.1 pct V) which meets the GM 980X specification.\*<sup>2</sup> It is produced by annealing in the intercritical

\*Yield strength, 0.2 pct offset, 380 MPa (55 ksi) max.; yield strength, 3.0 pct offset, 480 MPa (70 ksi) min.; tensile strength, 620 MPa (90 ksi) min.; elongation in 50 mm, 27 pct min.

( $\alpha + \gamma$ ) region (for example, ~three minutes at 788 °C) followed by air cooling to room temperature. The microstructural changes accompanying the mechanical property changes were studied by transmission electron microscopy, and this information was used to explain the observed mechanical property changes. This is the first known investigation of the tempering mechanisms in microalloyed dual phase steels. Understanding such mechanisms will certainly enable more efficient utilization of these new steels. The response of other dual phase steels to tempering might be different, being influenced by composition, the ratios of ferrite-martensite-retained austenite, and variations in processing parameters, and will have to be investigated.

## II. EXPERIMENTAL PROCEDURE

Standard (ASTM E8) tensile specimens were machined from sheet stock with the rolling direction parallel to

the specimen axis. Several specimens were heated for 60 minutes in a furnace at either 200, 300, 400, or 500 °C and air cooled to room temperature. Duplicate specimens representing each tempering condition were tension tested to failure at room temperature on an Instron testing machine at a crosshead speed of 5 mm per minute. Strain was measured with an Instron extensometer over a 50 mm gage length. Strain measurements were made from load-elongation data which was automatically plotted on a strip chart recorder.

Transmission microscopy specimens were obtained from 30 mm × 12.5 mm sections cut from the gage section of tempered, undeformed tensile specimen blanks and then surface ground to 0.625 mm using flood cooling. The ground piece was chemically thinned to 0.125 mm in a stirred and cooled solution containing 5 pct HF in H<sub>2</sub>O<sub>2</sub> (30 pct concentration). Discs 3 mm in diameter were electrodischarge machined from the chemically thinned sheet, mounted on a flat metal block with Scotch tape, and hand ground on both sides to 0.05 mm on 600 grit SiC paper. The ground discs were cleaned in acetone and alcohol and electropolished at room temperature in a Fischione "Twin-Jet" electropolishing unit in a solution of chromium trioxide, glacial acetic acid, and distilled water.

The foils were examined in a JEOL (JEM 200C) transmission electron microscope at an acceleration voltage of 200 kV. In addition to the conventional methods of transmission electron microscopy, such as bright field (BF), dark field (DF), and selected area diffraction (SAD), the weak beam dark field (WBDF) technique was also employed to enable resolution of the very fine precipitates and the interactions between precipitates and dislocations.

For estimating the retained austenite volume fraction, both linear intercept method using several low magnification dark-field micrographs of retained austenite as well as the frequency of occurrence of austenite in several high magnification micrographs were used.

## III. RESULTS

The stress-strain curve for the as-received steel was characteristic of dual phase steels (Figure 1), and the mechanical properties were superior to the minimum specified for GM 980X.<sup>2</sup> On tempering the steel above room temperature the yield point elongation ( $e_p$ ) appeared and various changes in mechanical properties resulted. Mechanical properties of the steel before and after tempering are listed in Table I and are also plotted in Figure 3 as a function of tempering temperature. The average absolute changes in strength and ductility were calculated from the preceding data and are also listed in Table I.

Minimal strength and ductility changes were observed on heating the steel at 200 °C. The yield strength (YS) increased slightly but the opposite was observed in the ultimate tensile strength (UTS); the total elongation ( $e_T$ ) and uniform elongation ( $e_u$ ) did not change. However, larger changes were observed on heating above 200 °C. Yield strength increased with increasing heating temperature and reached a maximum value at about 400 °C. The UTS decreased continually with increasing heating temperature and tended to level off at the higher temperatures.

The  $e_T$  and  $e_u$  were constant with tempering temperature up to 300 °C and dropped rapidly with higher tempering

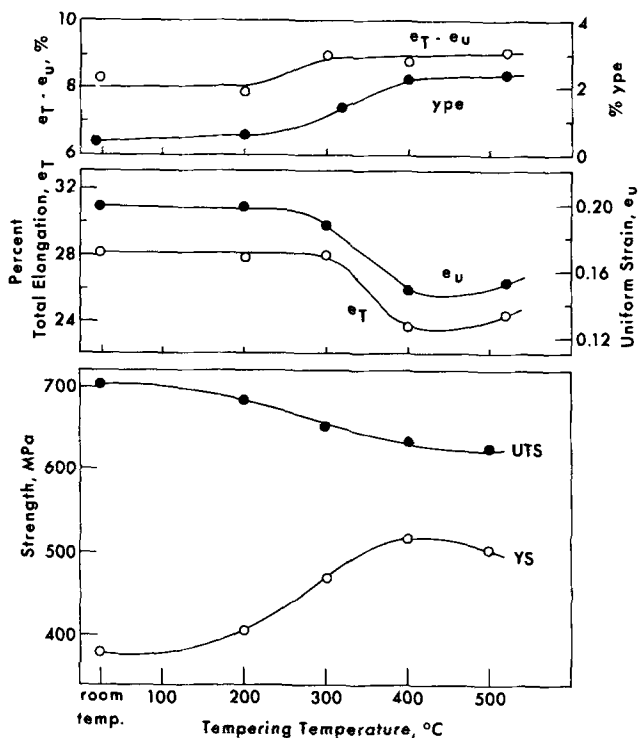


Fig. 3—Effect of tempering temperature on the mechanical properties.

temperatures. The combined reduction in UTS and elongation after the 400 °C tempering suggests a reduction in the toughness of the steel.

The post uniform elongation ( $e_T - e_u$ ), and ype are also plotted in Figure 3 as a function of tempering temperature. The ( $e_T - e_u$ ) was only minimally higher at tempering temperatures above 300 °C compared to the values in the as-received steel. The ype appeared on heating at 200 °C and increased in direct relation to heating temperature: it was 1.4 pct on heating at 300 °C and reached a maximum of about 2.4 pct on heating at higher temperatures, suggesting a rate dependent behavior. These maximum changes in ype correspond to similar limiting changes observed in strength and ductility on heating above 400 °C.

Earlier transmission electron microscopy of the same steel<sup>3</sup> showed that the as-received steel consisted of transformed and retained ferrite, martensite, and retained aus-

tenite. Transformed ferrite is ferrite that nucleates out of the austenite that forms during continuous annealing. With the exception of a small volume fraction of undissolved carbonitrides, the untransformed or retained ferrite was essentially free of fine vanadium carbonitride precipitates and contained a high density of accommodation dislocations, while the transformed ferrite contained banded precipitates and a lower density of dislocations, some of which were pinned by the precipitates. Substantial substructural twinning was found in the predominantly plate martensite. The retained austenite was principally in the form of 2 to 6  $\mu\text{m}$  diameter particles, although some submicron size particles were also present.

In conjunction with the minimal changes produced in the mechanical properties after tempering at 200 °C, minimal changes were also observed in the ferrite except for relaxation of residual stresses at the retained ferrite-martensite interfaces. The latter was inferred from the observation that many of these interfaces were cleaner and that they revealed less number of bend extinction contours at the interface in the tempered specimens as compared to the untempered specimens. Some rearrangement of the highly heterogeneous dislocation distributions into uniform low energy configurations was also noted. However, extensive twin boundary cementite particles were found in the martensite (Figures 4(a) and (b)). The cementite was in the form of thin platelets or films and is indicated by the arrows in Figure 4(c). Figure 4(c) was obtained from a superimposed cementite and double diffracted spot as revealed by the selected area diffraction pattern (Figure 4(d)). Diffraction patterns obtained from other martensite particles showed effects indicative of carbon clustering prior to precipitation. Minimal microstructural changes were observed in the retained austenite except for some very fine precipitates which were suspected to be carbides.

On tempering at 300 °C, a larger density of fine precipitates (< 2.5 nm in diameter) was found in both ferrites (for example, as seen in the retained ferrite in Figures 5(a) and (b)) compared to that found by the lower tempering treatment. Weak beam dark field analyses of some areas indicated that the precipitates were dislocation nucleated. These precipitates were suspected to be vanadium carbonitrides, but strong diffraction evidence could not be obtained. Also, some coarsening of the banded carbonitrides of the transformed ferrite was observed.

Table I. Effect of Tempering for One Hour on Mechanical Properties

| Tempering Temperature °C | Tensile Properties |         |           |           |                 |         |                 | Average Change in Properties |                       |                       |                             |                    |  |
|--------------------------|--------------------|---------|-----------|-----------|-----------------|---------|-----------------|------------------------------|-----------------------|-----------------------|-----------------------------|--------------------|--|
|                          | YS MPa             | UTS MPa | $e_T$ Pct | $e_u$ Pct | $e_T - e_u$ Pct | ype Pct | $\Delta$ YS MPa | $\Delta$ UTS MPa             | $\Delta$ (Pct $e_T$ ) | $\Delta$ (Pct $e_u$ ) | $\Delta$ (Pct $e_T - e_u$ ) | $\Delta$ (Pct ype) |  |
| AR                       | 390                | 699     | 28.1      | 19.9      | 8.2             | 0.2     |                 |                              |                       |                       |                             |                    |  |
|                          | 386                | 707     | 28.3      | 19.9      | 8.4             | 0.2     |                 |                              |                       |                       |                             |                    |  |
| 200                      | 400                | 683     | 28.5      | 20.0      | 8.5             | 0.5     |                 |                              |                       |                       |                             |                    |  |
|                          | 409                | 685     | 27.1      | 19.8      | 7.3             | 0.7     | + 16            | -19                          | -0.4                  | 0.0                   | -0.4                        | +0.4               |  |
| 300                      | 474                | 660     | 28.9      | 19.5      | 9.9             | 1.5     |                 |                              |                       |                       |                             |                    |  |
|                          | 462                | 647     | 27.0      | 18.5      | 8.5             | 1.3     | + 80            | -50                          | -0.2                  | -0.9                  | +0.7                        | +1.2               |  |
| 400                      | 521                | 637     | 22.8      | 14.8      | 8.0             | 2.2     |                 |                              |                       |                       |                             |                    |  |
|                          | 512                | 629     | 24.6      | 15.0      | 9.6             | 2.3     | +128            | -70                          | -4.5                  | -5.0                  | +0.5                        | +2.1               |  |
| 500                      | 499                | 612     | 24.8      | 15.8      | 9.0             | 2.5     |                 |                              |                       |                       |                             |                    |  |
|                          | 508                | 622     | 24.0      | 14.8      | 9.2             | 2.3     | +115            | -86                          | +3.8                  | -4.6                  | +0.8                        | +2.2               |  |

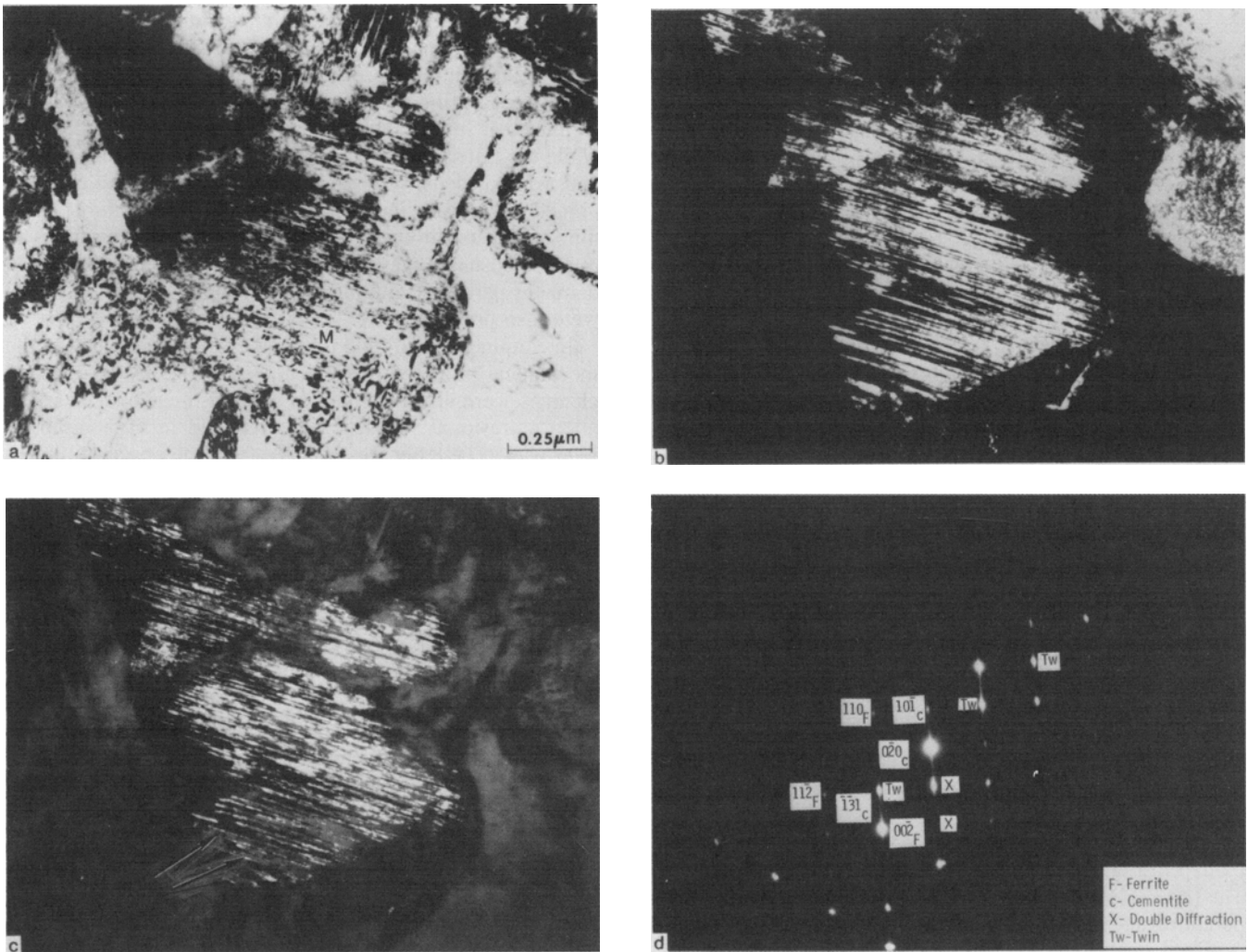


Fig. 4—Bright field transmission electron micrograph of a martensite particle in steel tempered at 200 °C (a), and (b) dark field micrograph of the same area. The dark field micrograph from superimposed cementite, double diffracted and streak from twinning reflections is shown in (c) and the corresponding indexed selected area diffraction pattern in (d) revealing substructural twinning and twin boundary cementite precipitation. Arrows in (c) point to the twin boundary cementite films, and the reflection used to form (c) is marked by aperture in (d).

Coarser cementite platelets (about 50 nm thick by 200 nm long) were found in the martensite, M in Figure 5(a), compared to the thin films observed after the 200 °C tempering. The substructural twinning in the martensite was no longer evident, indicating migration of the twin boundaries. About 90 vol pct of the retained austenite remained unchanged (Figure 5(c)) and was similar to that in the as-received steel while the remainder seemed to transform to a bainitic product.

Corresponding to the observed maximum changes in mechanical properties, gross microstructural changes were observed in specimens tempered at 400 °C and above. A substantial increase was observed in the density of fine precipitates (< 5 nm diameter), and in the amount of grain boundary and subboundary nucleated cementite and the intragranular cementite platelets in the retained ferrite (Figures 6(a) and (b)). Some dislocation pinning by the precipitates suggested that secondary hardening might result from such interactions. Extensive coarse cementite particles were found in the martensite similar to and more pro-

nounced than those observed after tempering at 300 °C.

More than 90 pct of the retained austenite was decomposed and was replaced by an upper bainitic product consisting of ferrite laths and lath boundary stringers of continuous cementite platelets. The existence of upper bainite was confirmed by morphological data and orientation relationships. Figures 6(c) and (d) show a completely decomposed retained austenite particle with cementite platelets, while the inset in Figure 6(d) shows the indexed diffraction pattern for the cementite.

Yet more changes were observed after tempering at 500 °C. The vanadium carbonitride precipitates in both ferrites were coarser than observed after lower temperature tempering and the banded nature of precipitates in the transformed ferrite was no longer evident (Figure 7(a)). Some cementite platelets were observed at grain boundaries. Coarse, partially spheroidized cementite particles were observed in the martensite (Figure 7(b)), and all the retained austenite was decomposed. About half of the retained austenite decomposed to an upper bainitic product (Figures 8(a)

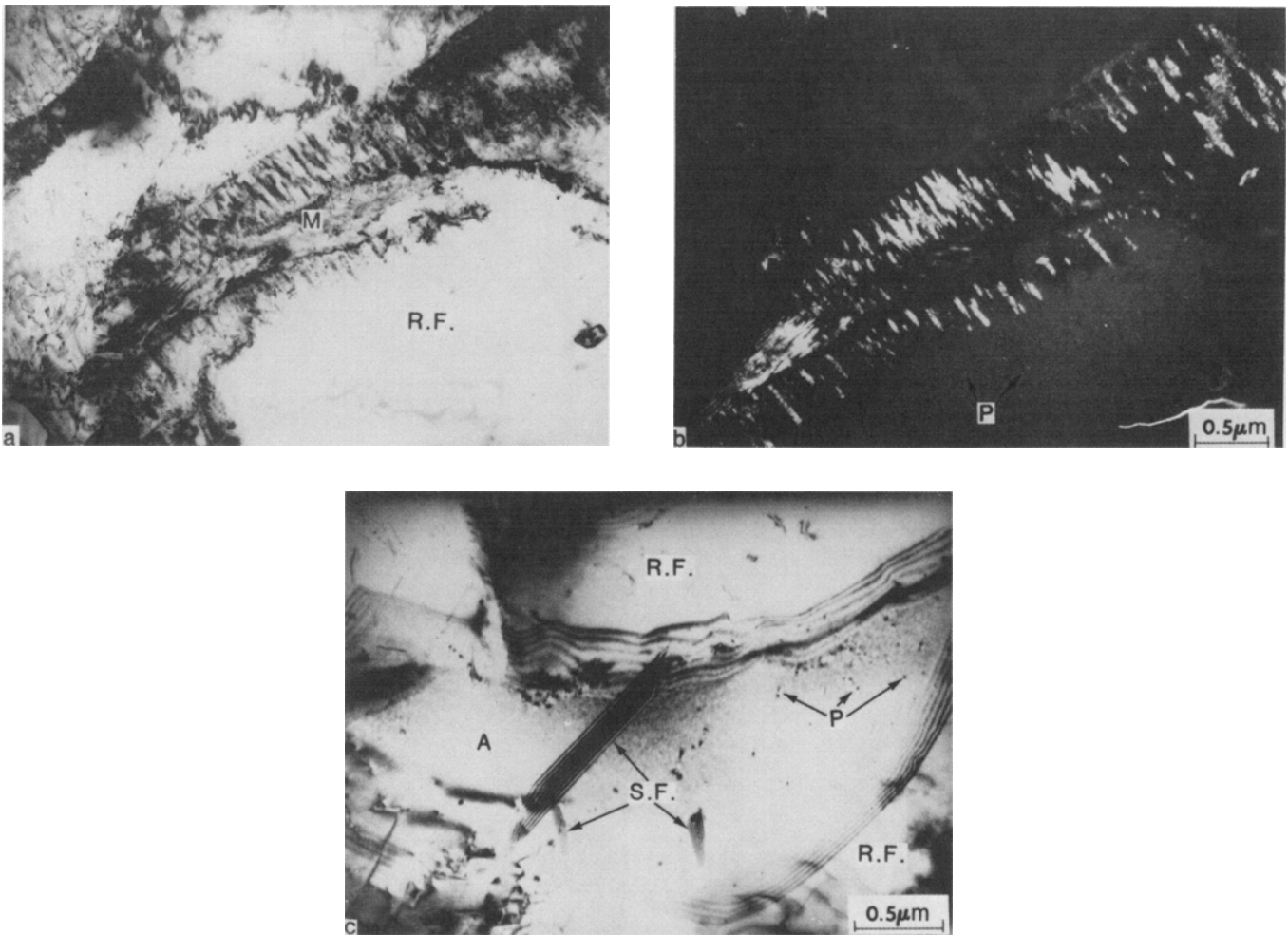


Fig. 5—Bright field (a) and dark field (b) micrographs revealing extensive cementite precipitation in a specimen tempered at 300 °C. 'P' in (b) shows extremely fine precipitation in the retained ferrite, R. F. (c) is the bright field micrograph from a different area showing the undecomposed retained austenite (A) following 300 °C tempering. S. F. reveals stacking faults and 'P', fine precipitation.

and (b)) similar to that observed after tempering at 400 °C. The remainder of the retained austenite decomposed by an interphase precipitation mechanism producing several ferrite-carbide morphologies including banded precipitation (Figure 8(c)).

#### IV. DISCUSSION

The mechanical properties of the steel were altered minimally on heating at 300 °C or below except for a small increase in YS and a similar decrease in UTS, the two latter changes indicating a corresponding decrease in the strain hardening rate of the steel. Heating above 300 °C decreased the UTS and ductility, however. These data suggest that the mechanical properties of this steel cannot be improved further by tempering below 500 °C.

The mechanical property changes produced by tempering seemed to be explainable in terms of the microstructural changes observed in the ferrite, martensite, and retained austenite. Tempering at 200 °C resulted in a slight increase in yield strength and a slight decrease in tensile strength. The yield strength increase may have been caused, in part, by the observed relaxation of residual stress at the ferrite-

martensite interfaces and rearrangement of dislocations in the ferrite. A part of the strength increase might also be caused by the fine precipitation that was observed in the retained austenite which could lead to higher mechanical stability in this phase.<sup>8,9,10</sup> The slight decrease in tensile strength must be related to the removal of solid solution carbon to precipitate twin boundary cementite particles in the martensite (Figures 4(a) and (b)). Such lowering of solid solution carbon is known<sup>11</sup> to soften the martensite.

The larger observed increase in yield strength and decrease in tensile strength after tempering at 300 °C compared to the 200 °C temper suggests an increase in the same microstructural changes noted at 200 °C, as was observed. The density of fine precipitates in both ferrites was higher as was the precipitation in the retained austenite resulting in the observed higher yield strength. As mentioned above, the latter would cause increased mechanical stability of the austenite. This could lead to a change in the mechanism of deformation induced transformation (TRIP) of retained austenite from one of stress induced to strain induced transformation. This change excludes the mechanical instability of the austenite triggering yield in the steel. The cementite precipitates in the martensite were coarser than at 200 °C

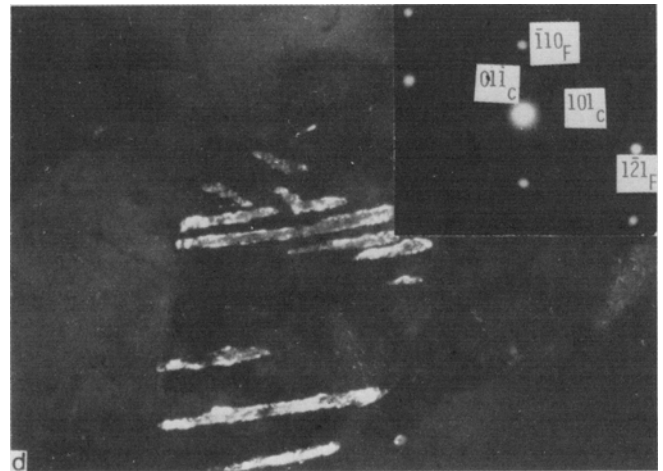
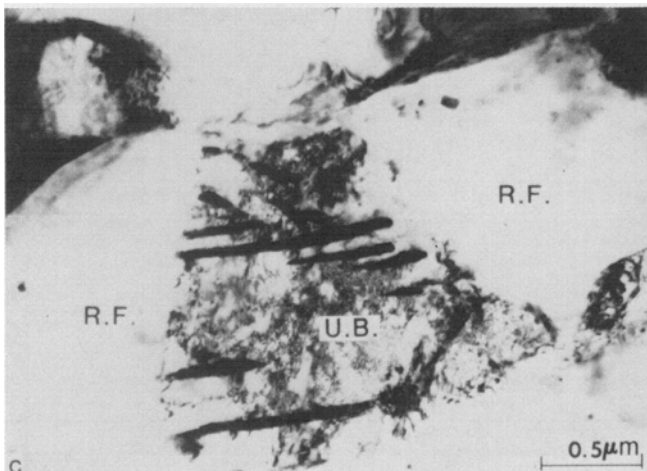
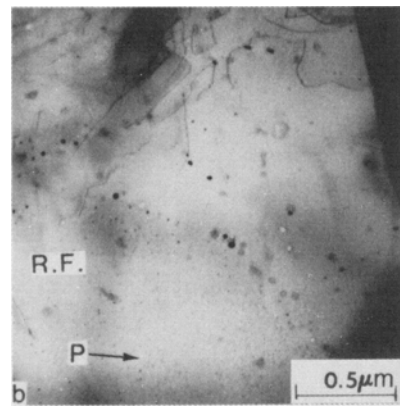
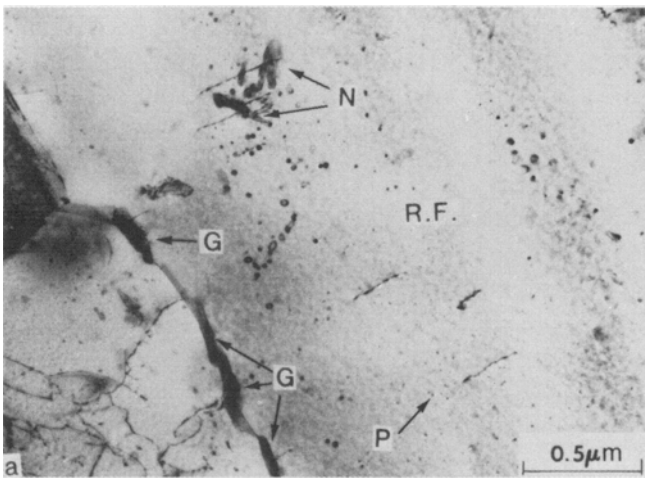


Fig. 6—Bright field micrographs (a) and (b) from 2 different areas of retained ferrite R. F. showing extensive fine precipitation, 'P', in a specimen tempered at 400 °C. In (a) 'G' shows intergranular cementite platelets and 'N', intragranular cementite platelets. Also seen in the 2 micrographs are several undissolved vanadium carbonitrides. Bright field (c) and cementite dark field micrographs (d) of the same area of 400 °C tempered specimen revealing the upper bainitic (U. B.) decomposition product of retained austenite. Inset in (d) reveals the indexed selected area diffraction pattern and the cementite reflections.

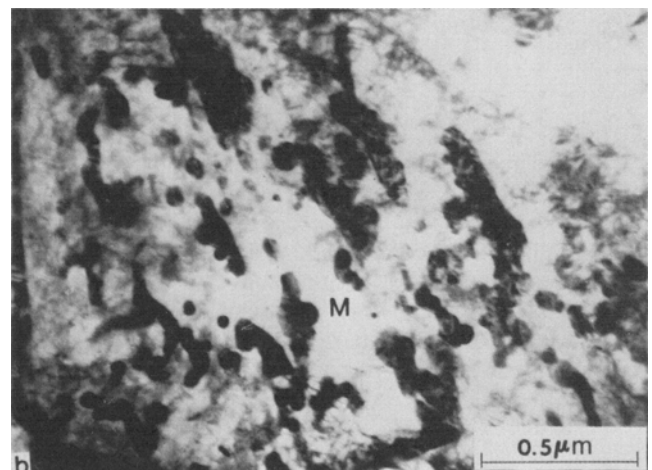
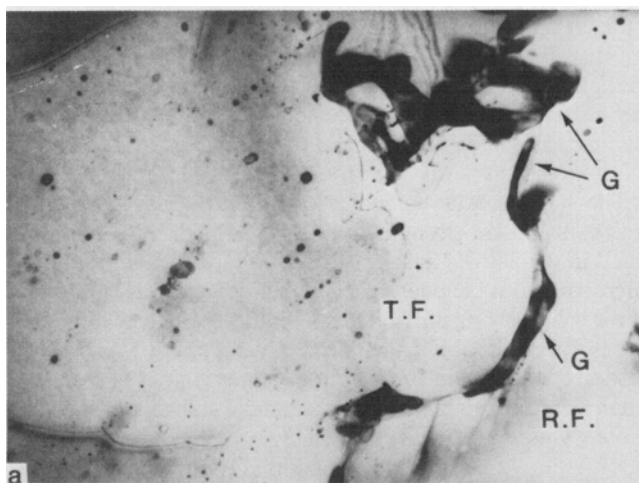


Fig. 7—Bright field micrographs from 2 different areas of the 500 °C tempered specimen revealing general coarsening of the alloy carbonitrides in the transformed ferrite, T. F., (a) and the early stages of spheroidization in the martensite, M, (b). 'G' in (a) shows intergranular cementite precipitation.

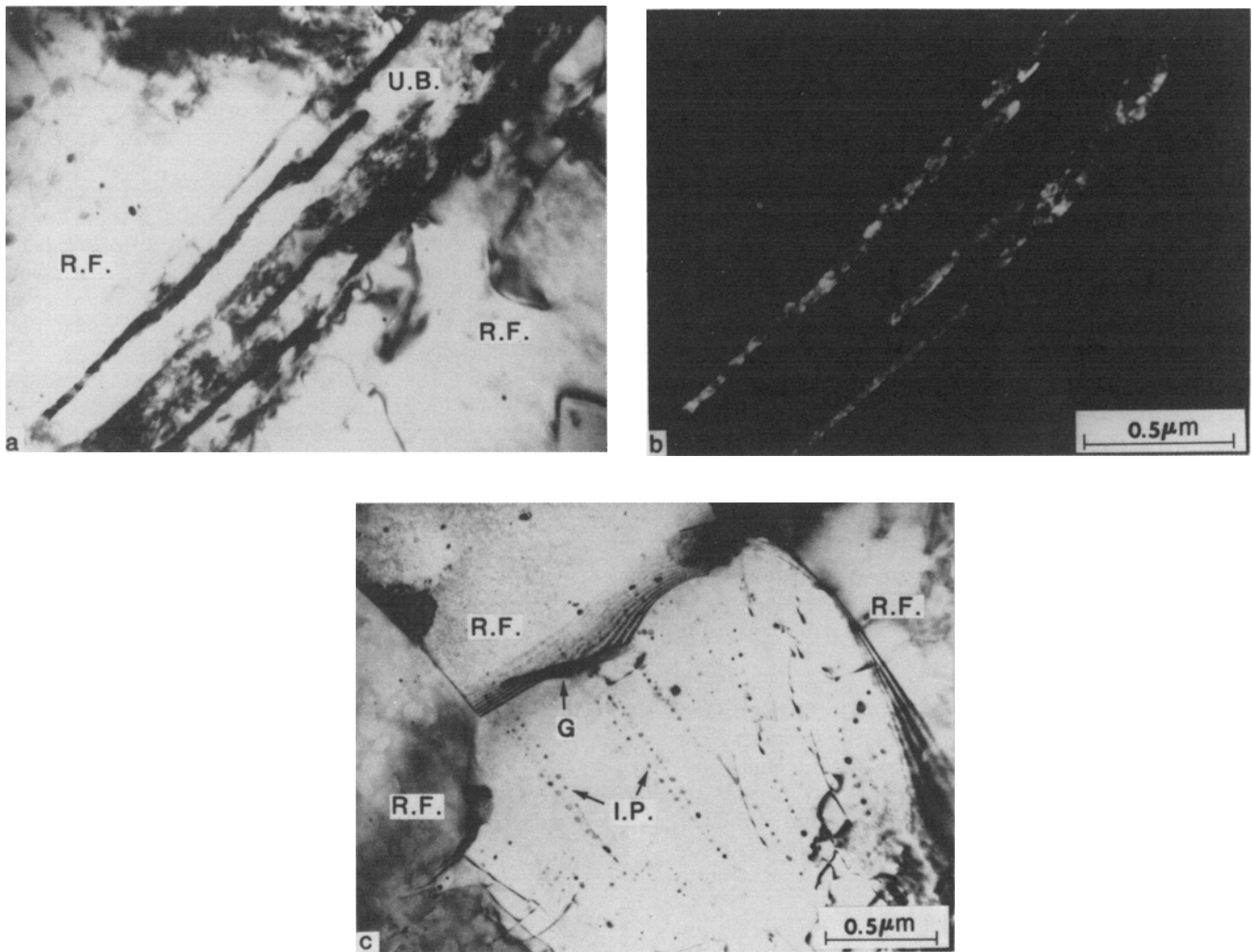


Fig. 8—Bright field (a) and cementite dark field micrographs (b) of the same area of 500 °C tempered specimen revealing upper bainitic (U. B.) decomposition product of retained austenite. (c) is the bright field micrograph from a different area of the same specimen revealing the interphase (I. P.) precipitation resulting from the decomposition of retained austenite. ‘G’ shows an intergranular cementite platelet.

and (Figures 5(a) and (b)) explain the larger decrease in UTS. These changes evidently affect  $e_T$  and  $e_u$  minimally. However, there was a slight increase in the ype suggesting an increase of dislocation-interstitial coupling.

Tempering at 400 °C resulted in a decrease in tensile strength and ductility, indicating an overall decrease in the toughness, as measured by the energy represented by the area under the stress-strain curve.

Extensive coarse cementite particles were found in the martensite, indicating continued progression of the tempering observed at lower temperatures. Such changes are known<sup>12</sup> to decrease strength but increase ductility. However, since both strength and ductility decreased in this steel, the decrease in ductility was attributed principally to the observed decomposition of retained austenite to upper bainite (Figures 6(c) and (d)) as discussed below.

Retained austenite is reported to influence dual phase steel strength and ductility. Upon deformation of the as-received steel the retained austenite transforms to martensite.<sup>13</sup> The transformation induced plasticity or TRIP enhances dual phase steel ductility<sup>7,14,3</sup> while the martensite

that forms upon deformation increases tensile strength. The actual TRIP mechanism(s) in dual-phase steels are far from being completely understood, and are quite likely<sup>7</sup> different from those of conventional “TRIP steels” which are made up entirely of a metastable austenitic microstructure.

The decomposition of retained austenite upon tempering eliminates the contribution from the TRIP mechanism leading to a lower strain hardening rate as evidenced by a lower  $e_u$  (Table I). In addition, the decomposition product of austenite upon tempering is upper bainite which has a lower strength compared to the transformation product of retained austenite due to deformation, *i. e.*, martensite. Thus, upon tempering in a temperature range wherein the retained austenite of the dual phase steel becomes thermally unstable and decomposes, the steel is expected to show a decrease in strength and ductility as was observed. A somewhat related embrittlement effect on properties due to tempering in the range 250 to 450 °C has been previously observed<sup>9,10</sup> in some structural steels. This embrittlement phenomenon which leads to lower toughness in structural steels is termed tempered martensite embrittlement. Tempered martensite

embrittlement in structural steels has been related<sup>9,10</sup> to both the decomposition as well as the morphology of decomposition product of retained austenite upon tempering. In contrast, in the dual phase steels where the emphasis is on strain hardening rate and formability, the decomposition and the consequent loss of retained austenite in the microstructure is more important than the nature of the decomposition product.

Transmission microscopy also produced evidence of dislocation nucleated precipitation of fine vanadium carbonitrides in the ferrite. This could cause "secondary hardening" and would explain some of the yield strength increase and the large ype observed. In addition, this fine precipitation could also reduce the flow properties of ferrite and a general lowering of ductility in ferrite.

The kinetics of martensite and austenite decomposition are faster at 500 °C and resulted in precipitate coarsening in the ferrite and coarsening of the cementite particles in the martensite. This was commensurate with the observed decrease in tensile strength. However, ductility increased and yield strength decreased slightly contrary to that observed after tempering at 400 °C. This seemed to be related to the unique decomposition observed in the retained austenite after this tempering treatment.

About half the retained austenite transformed to upper bainite similar to that observed at 400 °C, but the balance transformed to ferrite accompanied by the interphase precipitation of carbides, the latter microstructure being more ductile and with greater resistance to fracture than the former. In addition, there was some coarsening of the precipitation in both the ferrites which could facilitate dislocation motion. Consequently, somewhat higher ductility was observed on tempering at 500 °C.

In summary, tempering of this dual phase steel below 500 °C did not improve the tensile strength-uniform elongation combination. However, the response of other dual phase steels might be different. In this study the observed mechanical property changes produced by tempering below 500 °C could be explained reasonably well by the various microstructural changes that are produced by tempering. The operative mechanisms were precipitation in the ferrite and austenite, tempering of the martensite, and decomposition of the retained austenite. Different mechanisms dominated at different tempering temperatures and dictated the mechanical property changes. The largest decrease in ductility occurred on tempering above 400 °C and seemed to be related primarily to the decomposition of the retained austenite to upper bainite.

## V. CONCLUSIONS

1. The strength-uniform elongation combination of the as-received steel could not be improved by tempering below 500 °C.
2. The microstructure appears quite stable to heating at 200 °C, and minimal changes in mechanical properties

were observed. At higher tempering temperatures, tensile strength and ductility decreased with increasing temperature while yield strength increased and exhibited maximum values on heating above 400 °C.

3. At least four distinct microstructural changes seemed to occur during tempering, one or more being dominant in different tempering ranges. Precipitation of fine carbonitrides in the ferrite and austenite, and precipitation of cementite particles in the martensite seemed to govern mechanical properties for tempering treatments below 300 °C. Upon tempering above 400 °C, the decomposition of retained austenite appeared to have the principal influence on mechanical properties.
4. The largest loss in ductility (all in uniform elongation) occurred in heating the steel at 400 °C. At this tempering temperature, the steel also suffered a simultaneous loss in tensile strength. Concomitant with these mechanical property changes, the steel's microstructure revealed a nearly complete decomposition of retained austenite to an upper bainitic product and hence no contribution from the transformation induced plasticity or TRIP phenomenon resulting in the observed drop in uniform elongation.
5. Two competing mechanisms were identified for the decomposition of retained austenite on tempering at 500 °C, an upper bainitic transformation similar to that observed at 400 °C, and an interphase precipitation mechanism leading to ferrite with banded carbonitrides.

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