1974

Aging Characteristics of Aluminum-Silver Alloys

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AGING CHARACTERISTICS
OF ALUMINUM-SILVER ALLOYS

BY

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B.S.E., Florida Technological University, 1972

THESIS

Submitted in partial fulfillment of the requirements
for the degree of Master of Science in Engineering
in the Graduate Studies Program of
Florida Technological University

Orlando, Florida
1974

ACKNOWLEDGMENT

I wish to express my personal thanks to the many people concerned with the completion of my Master's Degree and Thesis.

Special thanks are extended to Dr. William F. Smith who, as the thesis director, gave his time to many helpful discussions and suggestions. Many thanks to the other members of the Committee in Charge, Drs. David R. Jenkins and Ernst M. Goldstein, for their very helpful advice and encouragement.

I further wish to express my special appreciation to my wife, Helen. Her understanding and patience with postponed plans and with many sleepless nights is gratefully appreciated along with her endless encouragement and assistance.

ABSTRACT

The effect of single-step and two-step aging treatments on the tensile properties of an Al-6 wt. % Ag alloy and an Al-14 wt. % Ag alloy has been investigated. Results show that the tensile properties of these alloys were approximately the same whether they were given single or two-step aging treatments. Reversion treatments applied to the fully age-hardened Al-14 % Ag alloy after 15 minutes at various temperatures between 200 and 350^{o}C showed two maxima in the ultimate tensile strength and the yield strength. It is believed that this result was caused by two transformation processes occurring simultaneously: i.e., (1) the coarsening and dissolution of G.P. zones and (2) the growth of the intermediate precipitate, γ'.

TABLE OF CONTENTS

INTRODUCTION

Age-hardening in the aluminum-silver alloy system has been studied extensively. Even though this alloy system is not the basis for any commercial aluminum alloys, it is of great value in the development of the theory of precipitation mechanisms. Two features of the aluminum-silver alloy system which make it particularly interesting are: (a) there is a large high temperature solid solubility (44.4 wt. % Ag at $566^{\circ}C$ [1]) with a rapid decrease in solid solubility with decreasing temperatures (0.75 wt. % Ag at $200^{\circ}C$ [1]), and (b) the size similarity in the aluminum and silver atoms. The basic sequence of aging in this system is classical in that it shows the normal sequence shown in most age-hardenable alloys, i.e., zones $\rightarrow$ intermediate precipitate $\rightarrow$ equilibrium precipitate.

Among the types of work that have been performed on this system are: hardness measurements [2-9], X-ray diffraction [10-29], electrical resistivity [30-36], electron microscopy [37-48], thermodynamic calculations [49,50], solubility studies [51,52] and tensile tests [53-59]. Most of this work has been comprehensively reviewed by Hardy and Heal [60] and by Kelly and Nicholson [61].

Kelly and Nicholson [61] have given the most probable aging sequence in the aluminum-silver alloys: Spherical silver-rich G.P. zones $\rightarrow \gamma' \rightarrow \gamma$ (Ag_2Al). Both the γ' and γ phases were believed to be hexagonal close packed by Nicholson and Nutting [39]. However, Hren and Thomas [41] thought they were a simple hexagonal crystal structure.

Many authors agreed that the γ' and the γ phases are precipitated from the face centered cubic aluminum rich matrix with the following orientation relationship:

$$(111)_{Al} \parallel (0001)_{\gamma' \text{ or } \gamma}, \quad [1\bar{1}0]_{Al} \parallel [11\bar{2}0]_{\gamma' \text{ or } \gamma} \qquad [39,61].$$

The general characteristics of the aging sequence are presented here and details will follow. Clusters of silver atoms are present at the solution heat treatment temperature (above 450°C [41]) and act as "nuclei" for G.P. zones [18]. The G.P. zones are spherical in shape in the aluminum-silver alloys due to the lack of a size difference between the silver and the aluminum atoms [20,62]. While aging isothermally the zones are growing competitively [41] and possibly, depending on time and temperature, there is a state change in the G.P. zones, from an ordered to a disordered state [24,28,52]. The volume fraction of zones remains the same [27,52] while the smaller G.P. zones dissolve supplying silver atoms to dislocations and stacking faults [39-41,43]. Soon these nucleation sites are strings, then rods, then platelets [61] of the metastable precipitate, γ'. Nicholson and Nutting [39] have given evidence that the plates contain stacking faults and upon aging the γ' becomes ordered and coarse.

Nucleation and Clustering of G.P. Zones

It was believed by Turnbull, Rosenbaum and Treaftis [35] and others [18,34] that aging starts during quenching, if the quench is slow. Furthermore, they believe that there is an increase in cluster size for slower quench rates. This fact is supported by Kähkönen [29]

who found that the size of the clusters is decreased and that aging is suppressed when very fast quench rates are used.

Walker, Blin and Guinier [14] have shown evidence that clustering of silver atoms occurs at solution treatment temperatures. Embryonic clusters are freely available from which larger clusters can develop during aging. According to Hren and Thomas [41], the silver-rich zones are observed in thin-foils of Al-20 wt. % Ag at temperatures greater than 450^{o}C, which is in the homogenization temperature range for these alloys. These zones (or clusters) formed at the high temperatures are responsible for the aging reported by Turnbull, et al. [35]

Walker and Guinier [18] have proposed from their X-ray diffraction data that the free energy of the aluminum-silver system is lowered when silver atoms surround themselves with other silver atoms. However, high temperatures (above the solid solubility limit) give rise to enhanced thermal motion which overcome any ordering due to the attraction of silver atoms for like atoms. They stated that this ordering may take place during the quench in the form of clusters of silver atoms. Because the clustering occurs so rapidly, they believe that a shell-like region was left with less than average silver content. This theory of a denuded region of silver around the G.P. zones has been shown to exist by actual composition measurements with X-ray small angle scattering techniques by Freise, Kelly and Nicholson [38] and by Baur and Gerold [52]. These researchers found that the denuded region was of zero to 0.2 at. % Ag, which is in good agreement with the theory.

Types of G.P. Zones

There are three types of G.P. zones that are found in the aluminum-silver alloys. These are the η', η and ε types which have been identified by Auer and Gerold [24] and substantiated by Yamaguchi and Ichimura [28]. The first type of G.P. zone, η', is found after quenching from above 380°C (which is the temperature derived from the metastable miscibility gap of Baur and Gerold [52]) to room temperature. The η'-type of G.P. zone remains f.c.c. containing 60 at. % Ag with a radius between 10 and 20 Å. The second, η, is found by aging below about 170°C and is also of f.c.c. structure and contains 55 at. % Ag. The radius of the η-zone increases with aging time or for higher concentrations of silver in aluminum. The third, the ε-type of G.P. zone is formed upon aging above approximately 170°C and is visible up to 350°C [41]. This zone contains only 30 at. % silver [24] because of a slightly larger zone radius [28,52].

Baur and Gerold [52] have given an interpretation which explains that the η-type zone has a more or less ordered arrangement of atoms within the zones, whereas in the ε-type there is a random distribution or clustering of silver atoms. Auer and Gerold [24] have further found that inhomogenities exist parallel to {111} -planes for the η' and ε-type and in the case of the η-type zones, they are parallel to the {100} -planes.

Dissolution of G.P. Zones

The most detailed research about the dissolution of the G.P. zones was reported by Hren and Thomas [41], who studied precipitation and dissolution in thin foils of an Al-20 wt. % Ag alloy in the hot stage of an electron microscope during aging. These authors found that the G.P. zones are very stable in the Al-Ag alloys upon aging except near growing γ' or near grain boundaries. Large zones grow at the expense of smaller ones and the volume fraction remains constant; that is, the zones grow competitively to very large sizes. Hren and Thomas [41] have found that the zones are stable up to approximately $350^{\circ}C$ where they then begin to dissolve. Upon reversion the zones decrease in diameter, first near growing γ' plates, where the silver atoms are taken up by the growing γ'. They have further observed that the γ' plates must dissolve before clustering may occur. This gives further evidence that clustering of the silver atoms must occur during the solution heat treatment.

Double Hardness Peaks

Many workers [5,21,28,39] have found that most Al-Ag alloys have hardness-aging time curves which exhibit clear double peaks when aged in the temperature range 100 to $200^{\circ}C$. They have shown that the double peaks are associated with G.P. zones and the γ' precipitate, respectively. Yamaguchi and Ichimura [28] have found by the aging of an Al-4 at. % Ag alloy at $140^{\circ}C$, the η' zone is transformed to the η zone which is responsible for the first peak. The nucleation and growth of γ' is believed

to be responsible for the second peak in hardness. They also found when aging at 200^{o}C and 300^{o}C of the same alloy the first peak is attributed to the ε- state and the second peak to the γ' peak.

The Growth of γ'

Many studies have been made on the nucleation and growth of the metastable precipitate, γ' and they are reviewed by Kelly and Nicholson [61]. Hren and Thomas [41] and Nemoto and Koda [43] agree that the nucleation of γ' is at Frank sessile and helical dislocations. However, Hren and Thomas [41] and Nemoto and Koda [43] showed that the nucleation of γ' is possible on all dislocations.

Barton and Ansell [44] show that there was a high density of helical dislocations and Frank sessile loops upon examination of the as-quenched solid solution. Thus there is no lack of sites for nucleation of γ' in Al-Ag alloys. Further, Frank, et al. [40] have shown evidence for precipitation at grain boundaries. They have shown that helical dislocations become straight near the boundaries by losing vacancies to the boundaries (the process of reverse climb). Owing to the effect of diffusion of silver atoms along dislocations, it can further be expected to find precipitates at grain boundaries.

It has been proposed that, as the helical dislocations are formed by the annihilation of vacancies at a screw dislocation [39], the local concentration of silver atoms around the helical dislocation is raised, making the helical dislocation a favorable site for the precipitation of a silver-rich phase. When this happens, it is

possible for stacking faults and Frank sessile dislocations to be produced. Then the stacking faults become enriched with silver atoms. The change from a silver-rich stacking fault or a helical dislocation to a thin γ' precipitate is continuous, with the silver atoms being supplied continuously by nearby dissolving G.P. zones [41]. The precipitate has an original shape of a rod, but then soon becomes thicker and wider into the shape of platelets. The γ' precipitate tends to grow in layers such that it has a general lenticular shape.

In its final stages of growth, the γ' phase was a hexagonal close-packed lattice [39,61]. However, it may be considered to have the same structure as the parent matrix, i.e., face centered cubic, but with a stacking fault on every other (111) plane. Nicholson and Nutting [39] also have proposed that, as the γ' plates grow larger with long aging times, the stacking faults are removed and the lattice becomes ordered.

During over-aging, the rapid growth of the γ' precipitates leads to coarsening [39]. As the partially coherent precipitate coarsens [37,39,61], it looses coherency with the matrix and this leads to pronounced softening [8]. Also, dislocations find it more favorable to avoid the precipitates rather than shear them, which also causes softening [39].

The Equilibrium Precipitate

The γ phase is the equilibrium precipitate formed by discontinuous precipitation. Reviews of the γ phase have been given by several authors such as: Aaronson and Clark [26], Laird and Aaronson [45,46], and Abbott and Haworth [47]. Nicholson and Nutting [39] have shown that γ nucleates discontinuously at grain boundaries. They have further indicated that this process does not require the nucleation of new grains, but instead the precipitation process is one of selective grain growth.

Strengthening Mechanisms in Alloys Containing Zones

Kelly [56] has given the theory of strengthening in an alloy containing zones and this has been further explained by many authors (Nicholson, Thomas and Nutting [37], Nicholson and Nutting [39], Price and Kelly [57,58], and Kelly and Nicholson [61]). Since Haessner and Schreiber [54] have shown that solid solution strengthening is small, and since the atomic radii of aluminum and silver differ by very little, lattice strains do not have an appreciable role in determining the strength of these alloys [56]. Kelly [56] has proposed that the strength be dependent primarily on the volume fraction of zones, the energy of the interface, and the Burgers vector of a dislocation intersecting the zone. To explain further, Kelly [56] has theorized when a dislocation passes through a solute-rich zone, the number of solute-solvent bonds is increased and this requires an increase in energy of the system. Consequently, a higher stress is needed to be

applied to force a dislocation through the zones than through the matrix. This effect is called chemical hardening [37,63].

Kelly [56] proposes that, provided the spacing of the precipitates is such that a dislocation passes through them as a rigid line, then the flow stress depends solely on the volume fraction of zones. He gives the equation:

$$\sigma = \frac{f^{\frac{1}{2}}\gamma}{\alpha\ b}$$

where σ is the flow stress, f is the volume fraction of zones, γ is the energy to shear a zone by one interatomic distance, b is the magnitude of the Burgers vector of the dislocation and α is a constant (for spherical particles it equals $2/\sqrt{\pi}$). However, some authors (Harkness, Gould, and Hren [27] and Baur and Gerold [52] have suggested and shown that the volume fraction of zones remains constant during aging, which would appear to give constant values for the flow stress. However, Kelly and Nicholson [61], imply indirectly that the flow stress might also be dependent, to some degree, on the particle size. However, the important point is that the strength of the alloy is dependent on the difficulty of dislocations to cut through the strong bonding in the solute-rich zones.

Strengthening Mechanisms In Alloys Containing γ'

Price and Kelly [58] used tensile tests on single crystals and observed multiple glide and very rapid work hardening rates in crystals containing γ'. They surmised that this was a result of

dislocations bypassing hard particles, but when this occurs dislocation loops are left at the particle. Thus, they believe that the very high work-hardening rates are caused by the interaction of the loops with subsequent glide dislocations. Cross slip of dislocations onto secondary slip planes is probably the cause of the observation of the multiple glide.

Barton and Ansell [44] examined tensile test samples using the electron microscope. They observed that the dislocation tangles were bounded by the precipitate platelets. Dislocations become more tangled and complex and the dislocations begin to move through the precipitates as the amount of deformation is increased. When the platelets of γ' are sheared, they are bent and twisted into shapes that are very different from their usual plate-like shape and flat appearance. During this stage, deformation is governed by the non-uniform interaction of dislocations with γ' platelets, other dislocations and with solute atoms.

RESEARCH OBJECTIVE

The purpose of this research was to examine the effects of different heat treatments on the tensile properties of two representative Al-Ag alloys. The different heat treatments were single-step, two-step and reversion aging treatments. The tensile properties investigated were the ultimate tensile strength and the yield strength along with the elongation of the samples.

EXPERIMENTAL PROCEDURE

Two compositions of the aluminum-silver system were examined. High purity Al-Ag alloys were used, one consisting of 6.0 wt. % silver and the other 13.9 wt. % silver (nominally Al-14 wt. % Ag). The actual chemical composition of these alloys is given in Table 1. Ingots of each of these were hot rolled to 0.125 inch sheets and then cold rolled to 0.025 inches using several intermediate annealing treatments. The 0.025 inch sheets were then sheared into 2.0 x 0.25 x 0.025 inch blanks and machined into tensile test specimens with a reduced section of 0.5 x 0.125 inches. Prior to all aging treatments, all of the samples were given solid solution heat treatments at 500^{o}C for 20 minutes in a

TABLE 1

CHEMICAL ANALYSIS OF SAMPLES USED*

a) Al-6 % Ag

Si	Fe	Cu	Mg	Ag
0.004 %	0.003 %	0.001 %	0.0002 %	6.0 %

Received from the Kaiser Aluminum Company

b) Al-14 % Ag

Si	Fe	Cu	Mn	Mg	Cr
0.001 %	0.001 %	0.002 %	<0.001 %	0.0007 %	<0.001 %

Ni	Zn	Ti	Ga	V	Ag
<0.001 %	<0.002 %	<0.001 %	<0.002 %	<0.001 %	13.9 %

Received from the Reynolds Metals Company

* All percentages in wt. %

furnace which was controlled to $\pm$ 3^{o}C. After the solution heat treatment, the samples were water quenched at 20^{o}C and aged in a bath of Ucon heat transfer fluid controlled to $\pm$ 1^{o}C.

Single-step, two-step, and reversion aging treatments were performed on both of these alloys to determine their characteristics under different aging conditions. Single-step aging treatments consisted of aging at 160 and 180^{o}C for various times for both alloys. An additional aging treatment at 140^{o}C was performed on the Al-14 wt. % Ag alloy. The two-step aging treatments consisted of aging at 80^{o}C for one week plus various times at 160^{o}C for both alloys. Another two-step aging treatment was performed in which the samples from both alloys were aged for 24 hours at 100^{o}C plus reaging for various times at 160^{o}C. Reversion experiments were accomplished by aging both compositions at 160^{o}C for 48 hours to a fully age-hardened condition, then 15 minutes at temperatures between 200 and 350^{o}C and water quenching again at 20^{o}C. A summary of the aging treatments performed is given in Table 2.

After the heat treatments, the samples were tested for ultimate tensile strength, 0.2 % offset yield strength and elongation using a 10,000 lb. Instron machine (0.1 inches per minute) and an Instron strain gage extensometer. All samples were tested at room temperature in duplicate. In cases where obvious discrepancies in the results occurred additional tests were made.

TABLE 2

SPECIMEN AGING TREATMENTS
Al-6 wt. % Ag and Al-14 wt. % Ag

All samples initially solution heat treated for 20 minutes at 500°C and water quenched at 20°C.

Single-Step	Two-Step	Reversion
a) Age at 140°C* b) Age at 160°C c) Age at 180°C for various times up to 120 hours.	a) Age at 80°C for one week then 160°C for various times from 1 to 95 hours. b) Age at 100°C for 24 hours then 160°C for various times from 4 to 48 hours.	Age at 160°C for 48 hours then 15 minutes at either: 200, 225*, 250, 275*, 300, or 350°C then water quenched at 20°C.

* Al-14 wt. % Ag alloy only.

RESULTS

One-Step Aging

(a) Al-6 % Ag Alloy

The effects of one-step aging treatments at 160° and 180°C on the tensile properties of the Al-6 % Ag alloy are shown in Fig. 1 and 2.* After one-step aging at 160°C, the ultimate tensile strength (UTS) of the Al-6 % Ag alloy increased almost linearly and attained a value of 23.5 ksi after 96 hr (Fig. 1). However, between 1/2 and 4 hr there is a decrease in the rate of strengthening and then a subsequent increase of strengthening. This change in strengthening rate is believed to be due to a phase transformation and will be discussed later.

On aging at 180°C for 96 hr the UTS was 21.4 ksi (Fig. 2) which was slightly lower than the value for 160°C aging. The yield strengths after 96 hr of aging were 15.2 ksi for the 160°C age and 13.2 ksi for the 180°C age. Thus in both cases, after aging for 96 hr, slightly higher strengths were obtained with the lower temperature aging treatment at 160°C. There is also an indication of the change in the rate of strengthening.

The elongation values for 8 of the 9 tests in this alloy aged at 160°C were centered aroung 20% with a deviation of about ± 2%. At 180°C the ductility showed a trend to decrease with elongation values dropping from 23.5% to 18% after 96 hr aging.

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* Actual test data for all tensile tests are listed in the appendix.

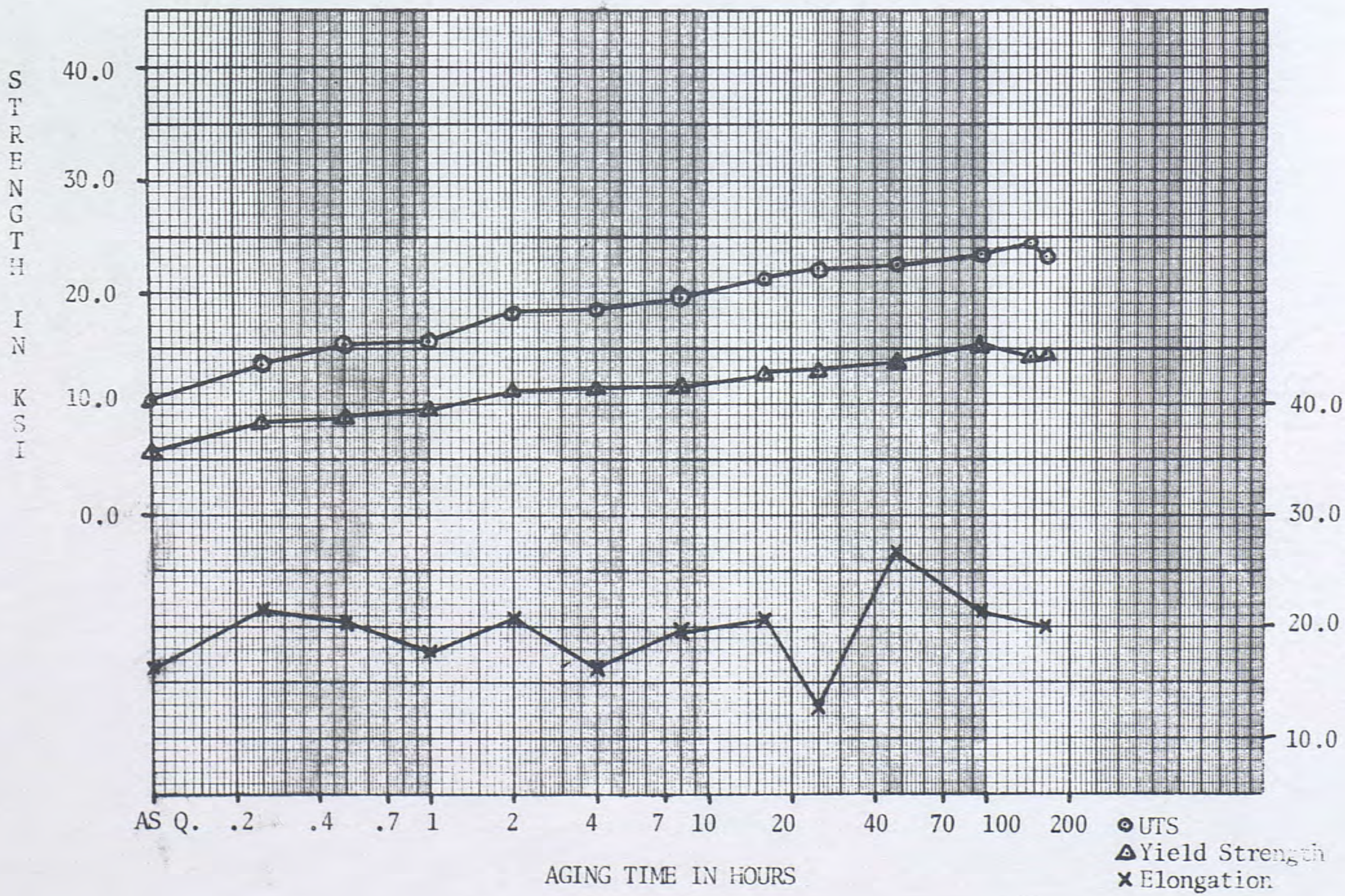

Fig. 1.--The effects of single-step aging treatments at 160°C on the tensile properties of the Al-6 wt. % Ag alloy.

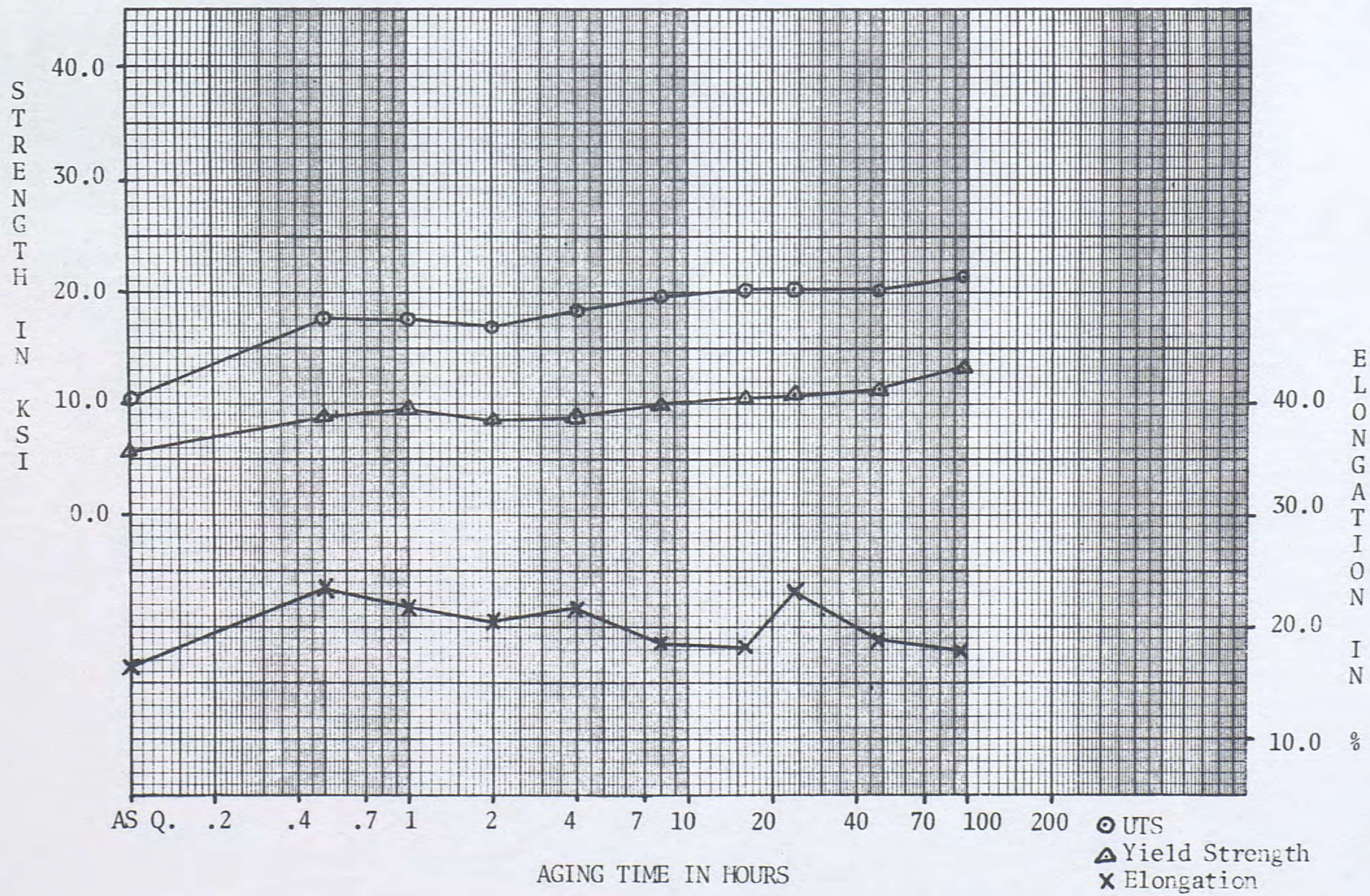

Fig. 2.--The effects of single-step aging treatments at 180°C on the tensile properties of the Al-6 wt. % Ag alloy.

(b) Al-14 % Ag Alloy

The effects of one-step aging treatments at 140, 160 and 180°C on the tensile properties of the Al-14 % Ag alloy are shown in Fig. 3, 4 and 5. After one-step aging at 140°C, the UTS of the Al-14 % Ag alloy increased almost linearly and obtained a value of 36.6 ksi after 96 hr (Fig. 3). Between one and two hours there is a decrease in the rate of strengthening and then a subsequent increase after two hours. This change is also thought to be due to a phase transformation.

On aging at 160°C for 96 hr the UTS was slightly lower reaching 35.7 ksi (Fig. 4). Samples aged at 180°C reached a maximum UTS of 36.9 ksi after 24 hr and then decreased in strength to 34.5 ksi after 96 hr (Fig. 5).

The yield strengths for 140, 160 and 180°C single-step aging treatments for the Al-14 % Ag alloy followed the same trend as the UTS but at a level about 8 to 12 ksi lower. After 96 hr of aging, the yield strengths were 26.3, 28.2 and 25.8 ksi for the 140, 160 and 180°C aging treatments respectively.

In the case of the 160°C aging treatment, the difference between the yield strength and the UTS gets smaller with long aging times. This indicates the material is becoming less ductile as is shown by the rapid decrease in the elongation values.

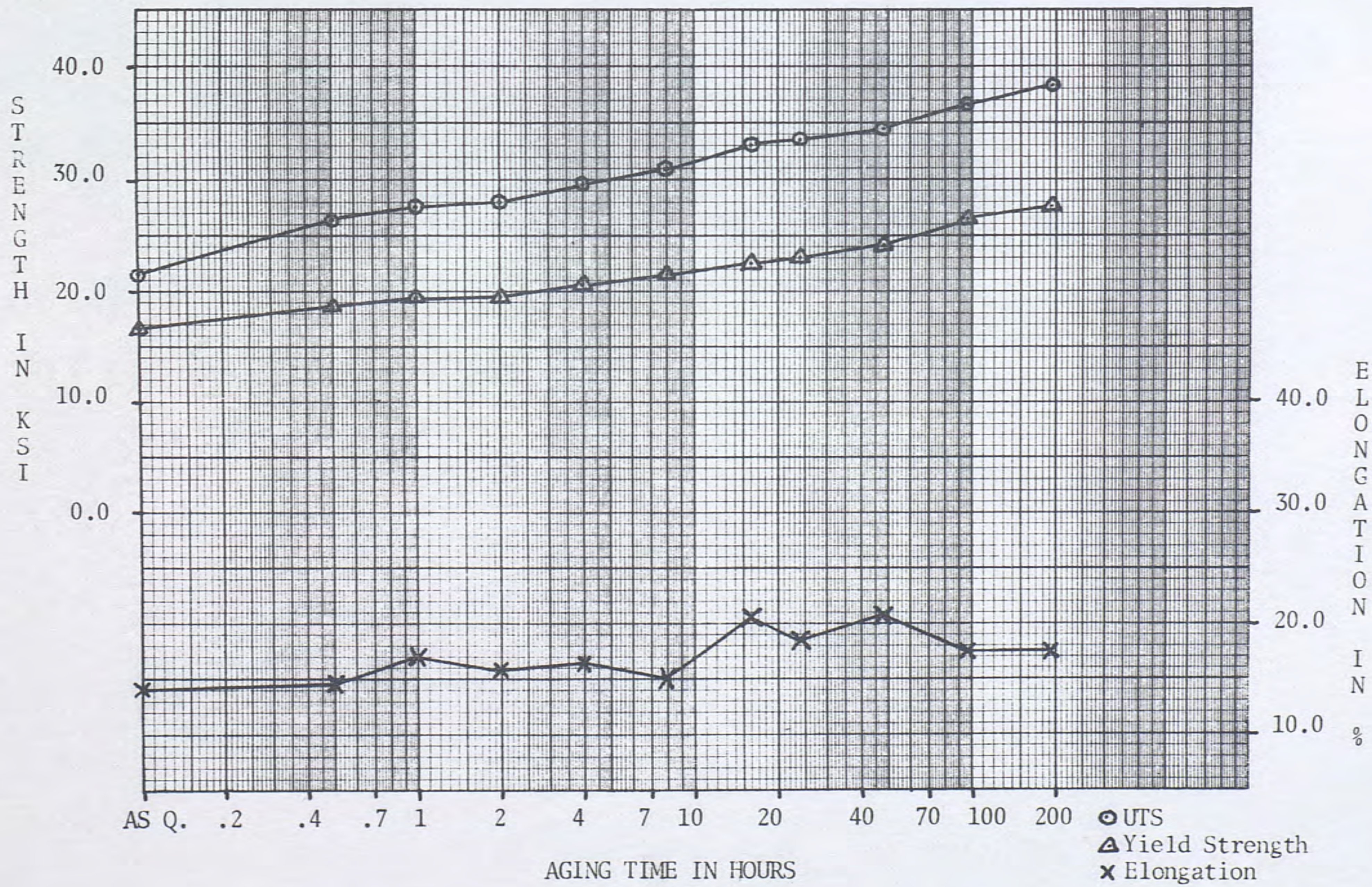

Fig. 3.--The effects of single-step aging treatments at 140°C on the tensile properties of the Al-14 wt. % Ag alloy.

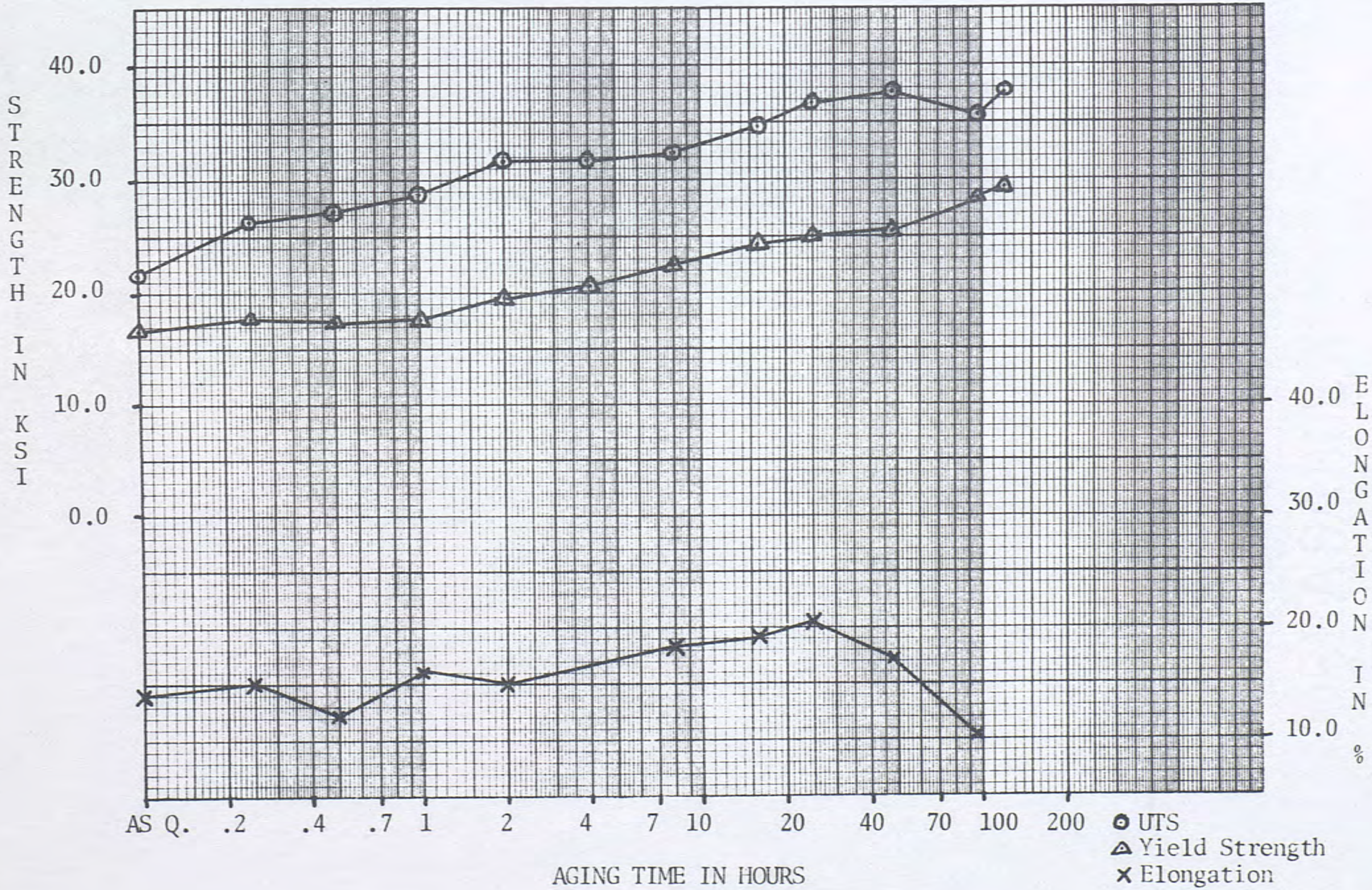

Fig. 4.--The effect of single-step aging treatments at 160°C on the tensile properties of the Al-14 wt. % Ag alloy.

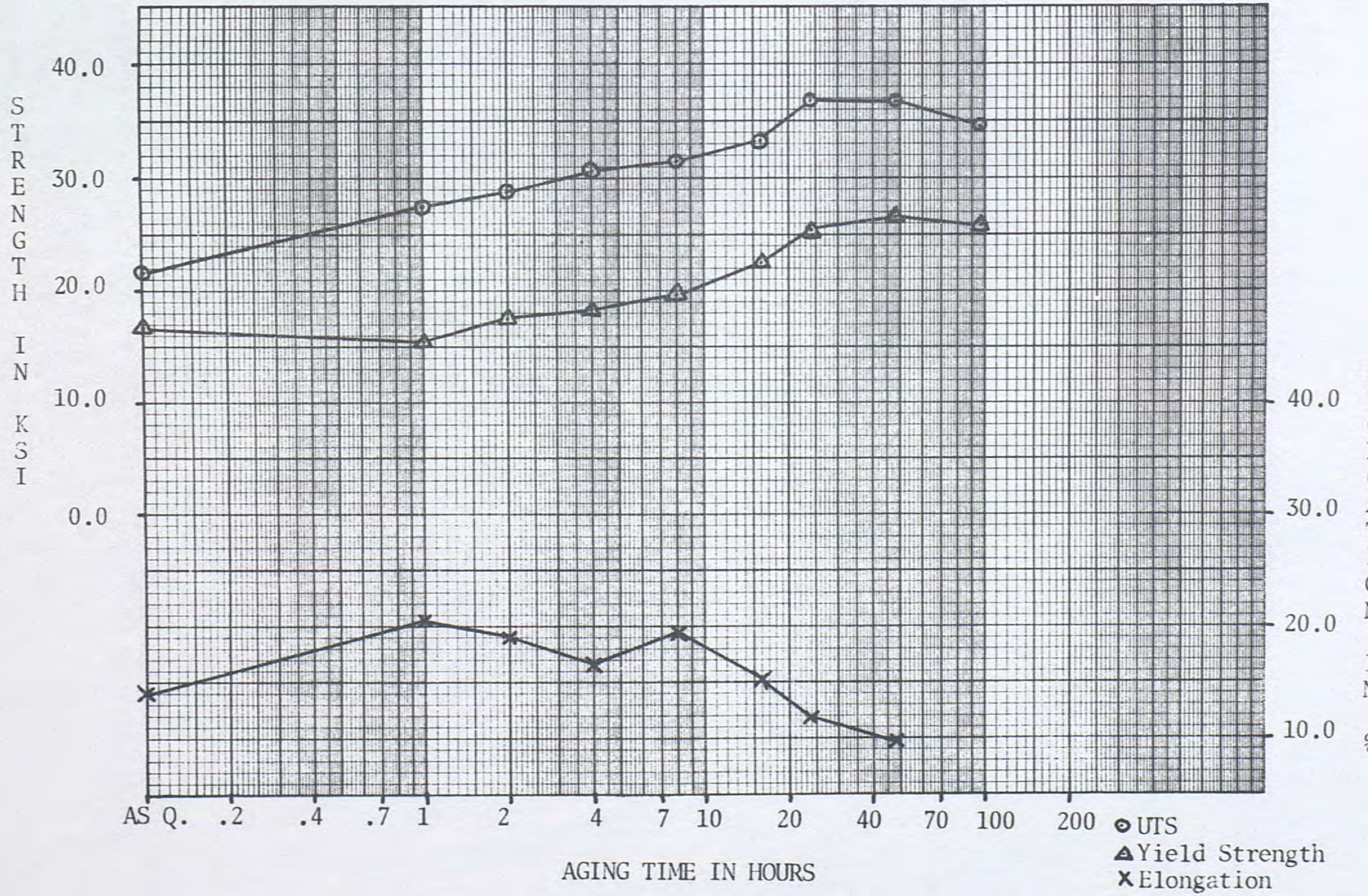

Fig. 5.--The effects of single-step aging treatments at 180°C on the tensile properties of the Al-14 wt. % Ag alloy.

Two-Step Aging

(a) Al-6 % Ag Alloy

After two-step aging for one week at 80°C and then aging at 160°C, the UTS attained a value of 24.0 ksi after 96 hr (Fig. 6). This value is almost identical to that obtained with single-step aging at 160°C for 96 hr which had a UTS of 23.5 ksi (Fig. 1). Therefore there was no significant change in the UTS values for this aging experiment.

It was observed that in the critical aging temperature range where a transformation is believed to occur, the UTS was an average of about 2 ksi higher for the two-step age compared to the one-step aging treatment. However, the yield strength was about 2 ksi lower after 96 hr for the two-step aging treatment.

The effects of two-step aging treatments for 24 hr at 100°C plus 160°C aging on the tensile properties of the Al-6 % Ag alloy are shown in Fig. 7. No significant differences were observed in the UTS or the yield strength when compared to the single-step aging treatment at 160°C for the Al-6 % Ag alloy.

(b) Al-14 % Ag Alloy

After two-step aging of the Al-14 % Ag alloy at 80°C for one week plus 160°C aging (Fig. 8) there appears to be no significant difference in the values of the UTS when the one-step and the two-step aging treatments are compared. But after the longer aging times, a higher strength was observed after about 100 hr with the difference in the UTS being 4.5 ksi. The yield strength values, however, were an

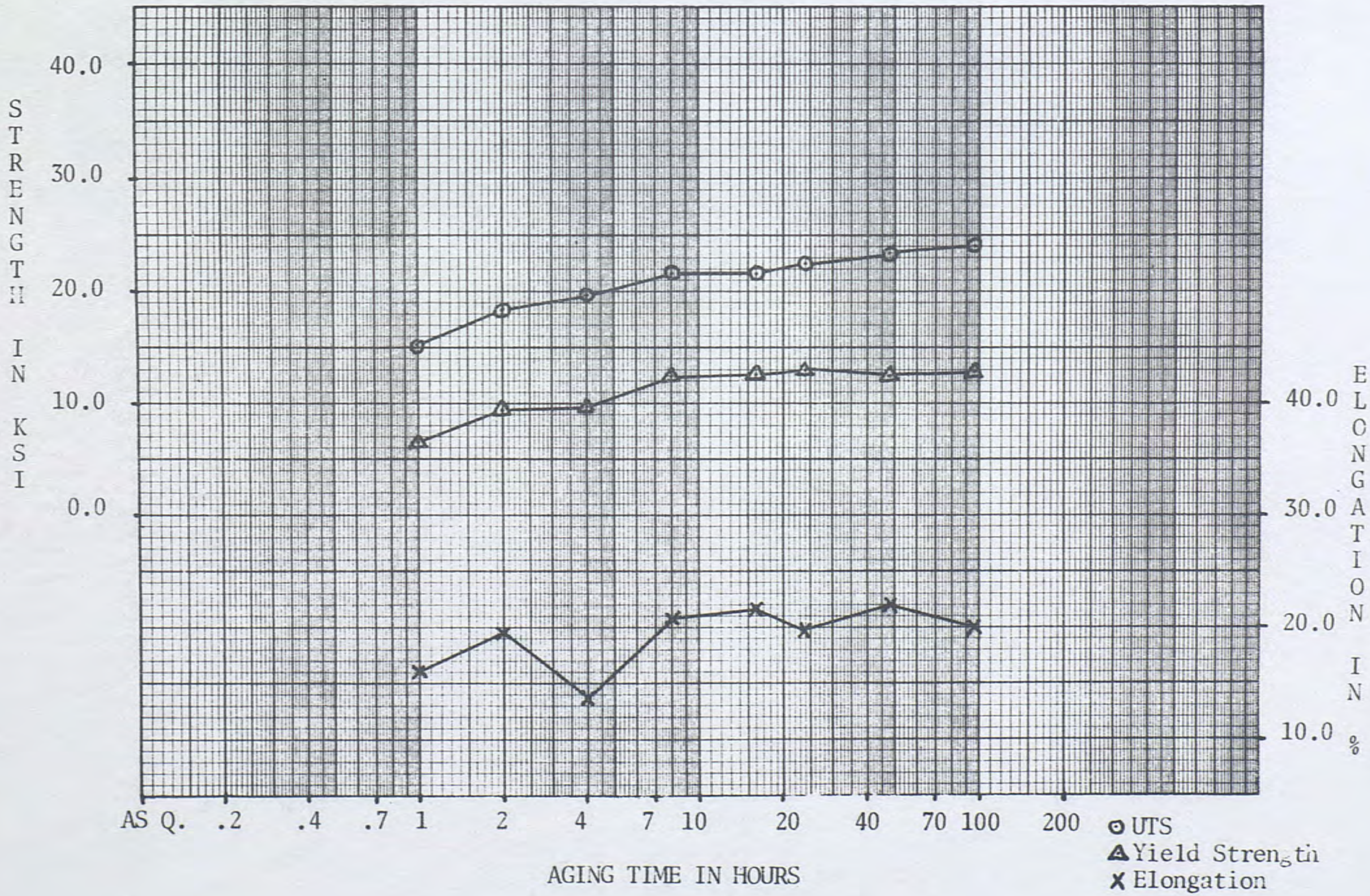

Fig. 6.--The effects of two-step aging treatments at 80°C for one week plus aging at 160°C on the tensile properties of the Al-6 wt. % Ag alloy.

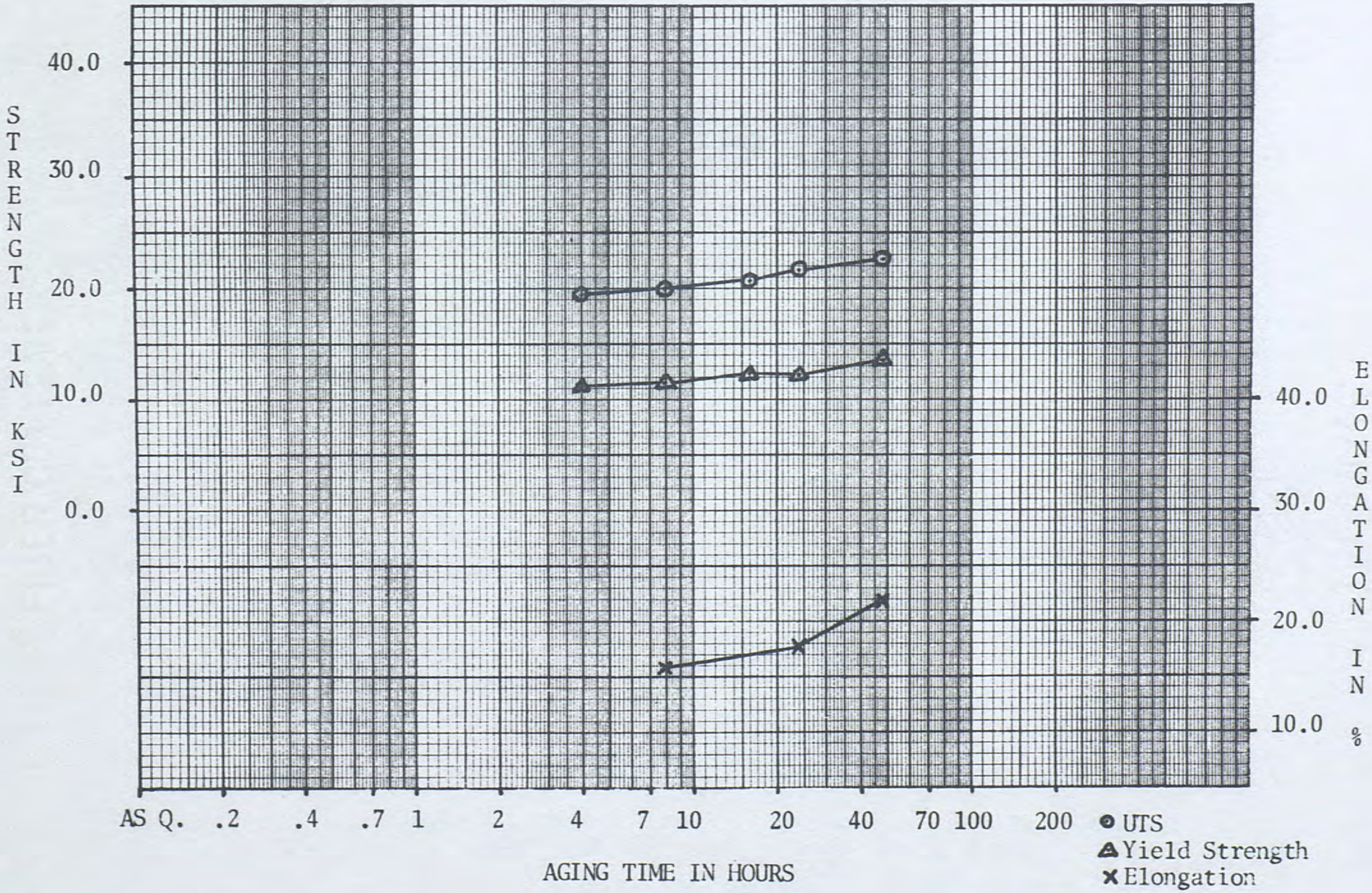

Fig. 7.--The effects of two-step aging treatments at 100°C for 24 hours plus aging at 160°C on the tensile properties of the Al-6 wt. % Ag alloy.

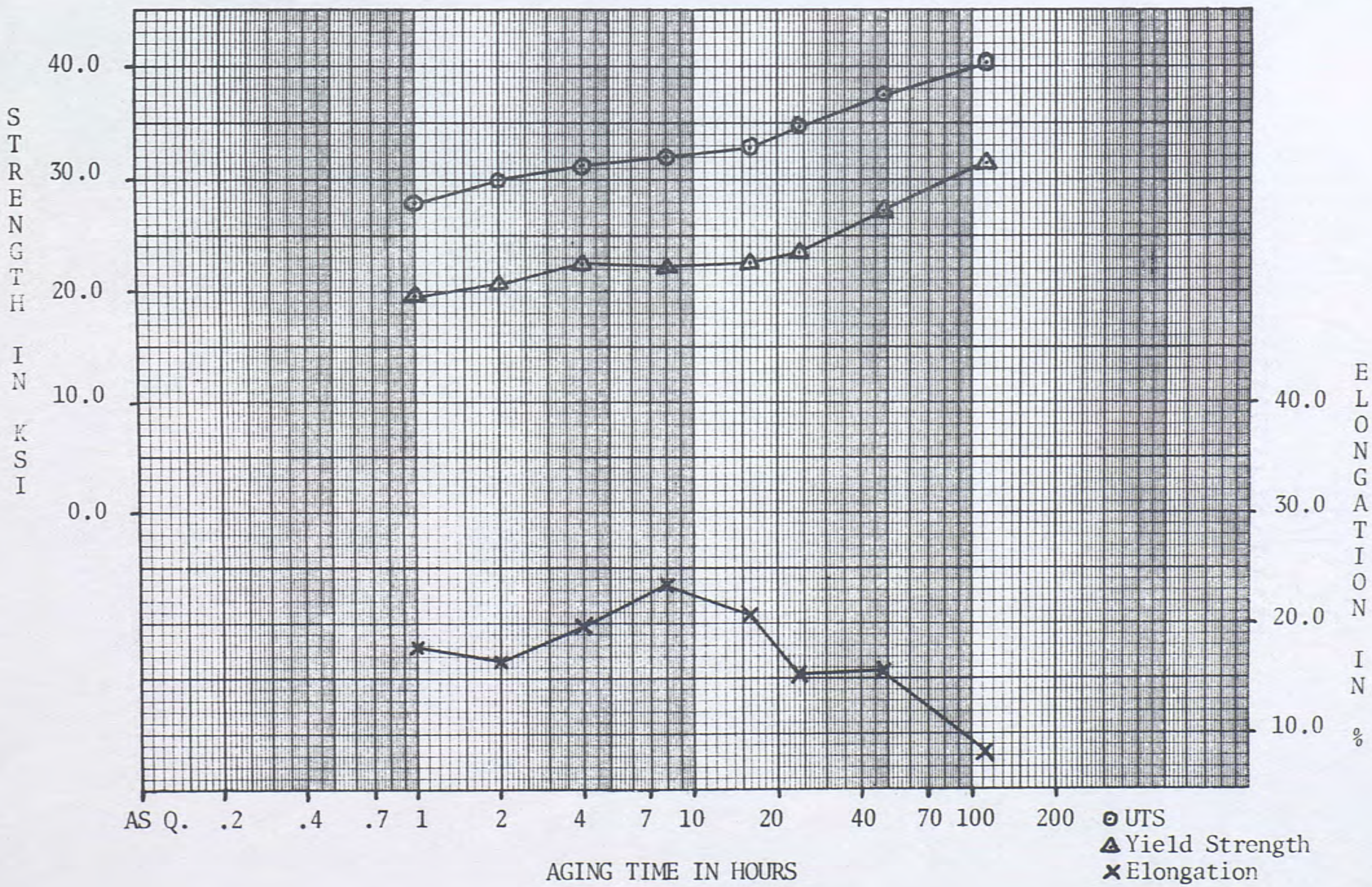

Fig. 8.--The effects of two-step aging treatments at 80°C for one week plus aging at 160°C on the tensile properties of the Al-14 wt. % Ag alloy.

average of 2 ksi higher for the two-stage aging treatment except between 8 and 24 hr where there is a change in the rate of strengthening and a decrease in the ductility.

The effects of two-step aging at 100°C for 24 hr plus 160°C aging on the tensile properties of the Al-14 % Ag alloy are shown in Fig. 9. The values for the UTS and the yield strength of the two-step aging treatment were almost the same as the values from the one-step aging at 160°C.

Reversion

(a) Al-6 % Ag

After first aging the alloy for 48 hr at 160°C the UTS was 22.5 ksi and the yield strength was 13.9 ksi. After 15 min at 200°C the UTS dropped to 19.2 ksi and after 15 min at 250, 300, or 350°C the UTS continued to decrease almost linearly (Fig. 10). The UTS for reversion for 15 min at 350°C was 10.8 ksi. This value was still higher than that of the sample quenched from solution heat treatment which had a UTS of 10.2 ksi.

The yield strength, however, did not show the same linear decrease (Fig. 10). There was an initial drop to 9.2 ksi after 15 min at 200°C and an increase to 10.3 ksi after 15 min at 250°C. Reverting the material for 15 min at 300 and 350°C then reduced the yield strength even lower and was accompanied with large increase in ductility.

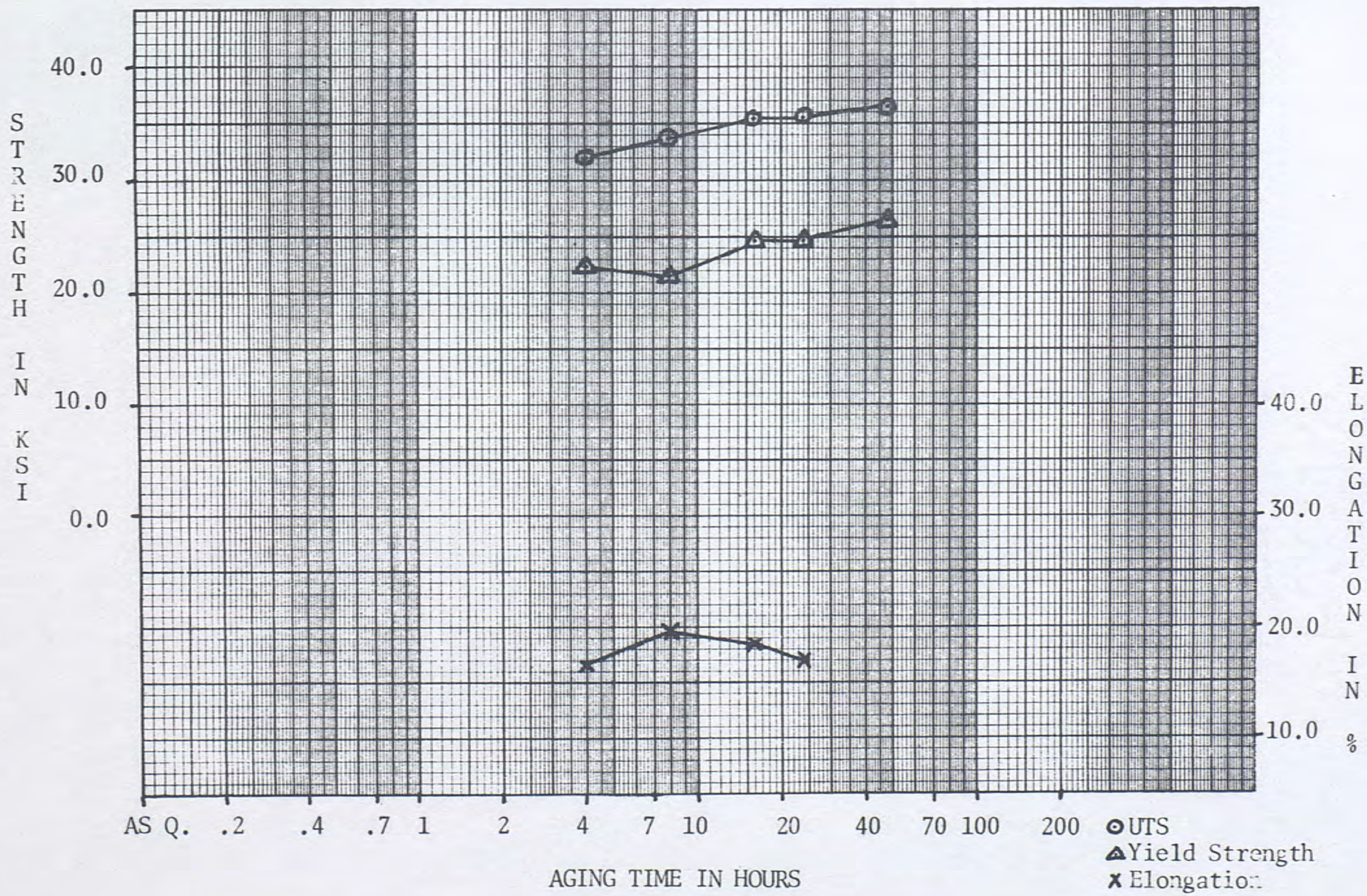

Fig. 9.--The effects of two-step aging treatments at 100°C for 24 hours plus aging at 160°C on the tensile properties of the Al-14 wt. % Ag alloy.

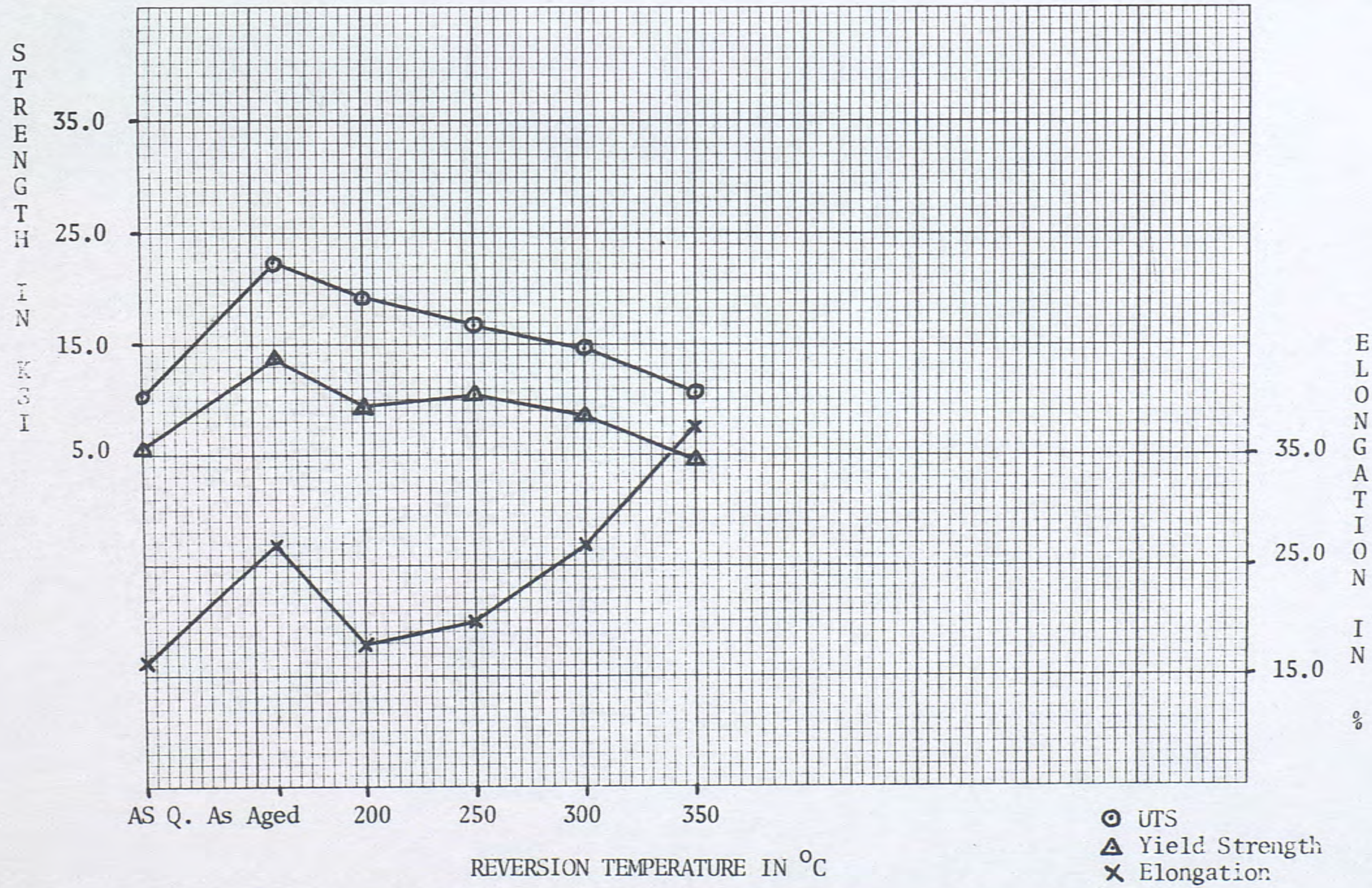

Fig. 10.--The effects of reversion treatments for 15 minutes at various temperatures on the tensile properties of a fully age-hardened Al-6 wt. % Ag alloy.

(b) Al-14 % Ag Alloy

The effects of aging for 48 hr at 160°C and then reversion for 15 min at either 200, 225, 250, 275, 300, or 350°C on the tensile properties of the Al-14 % Ag alloy is shown in Fig. 11. An initial decrease in strength followed by a subsequent increase and then by a second decrease after higher reversion temperatures is observed in both the UTS and the yield strengths of the Al-14 % Ag alloy (Fig. 11). This phenomenon was also observed in the yield strength of the Al-6 % Ag alloy upon reversion treatment to a much less degree.

The Al-14 % Ag alloy reaches a maximum UTS of 36.8 ksi and a yield strength of 26.5 ksi after a reversion treatment of 15 min at 250°C. These values represent a slightly lower UTS and a higher yield strength when compared to the values obtained from aging at 160°C for 48 hr (which were a UTS of 37.6 ksi and a yield strength of 25.5 ksi).

The UTS of 26.0 ksi after a reversion treatment of 15 min at 350°C is still higher than the sample quenched from solution heat treatment which has a UTS of 21.5 ksi.

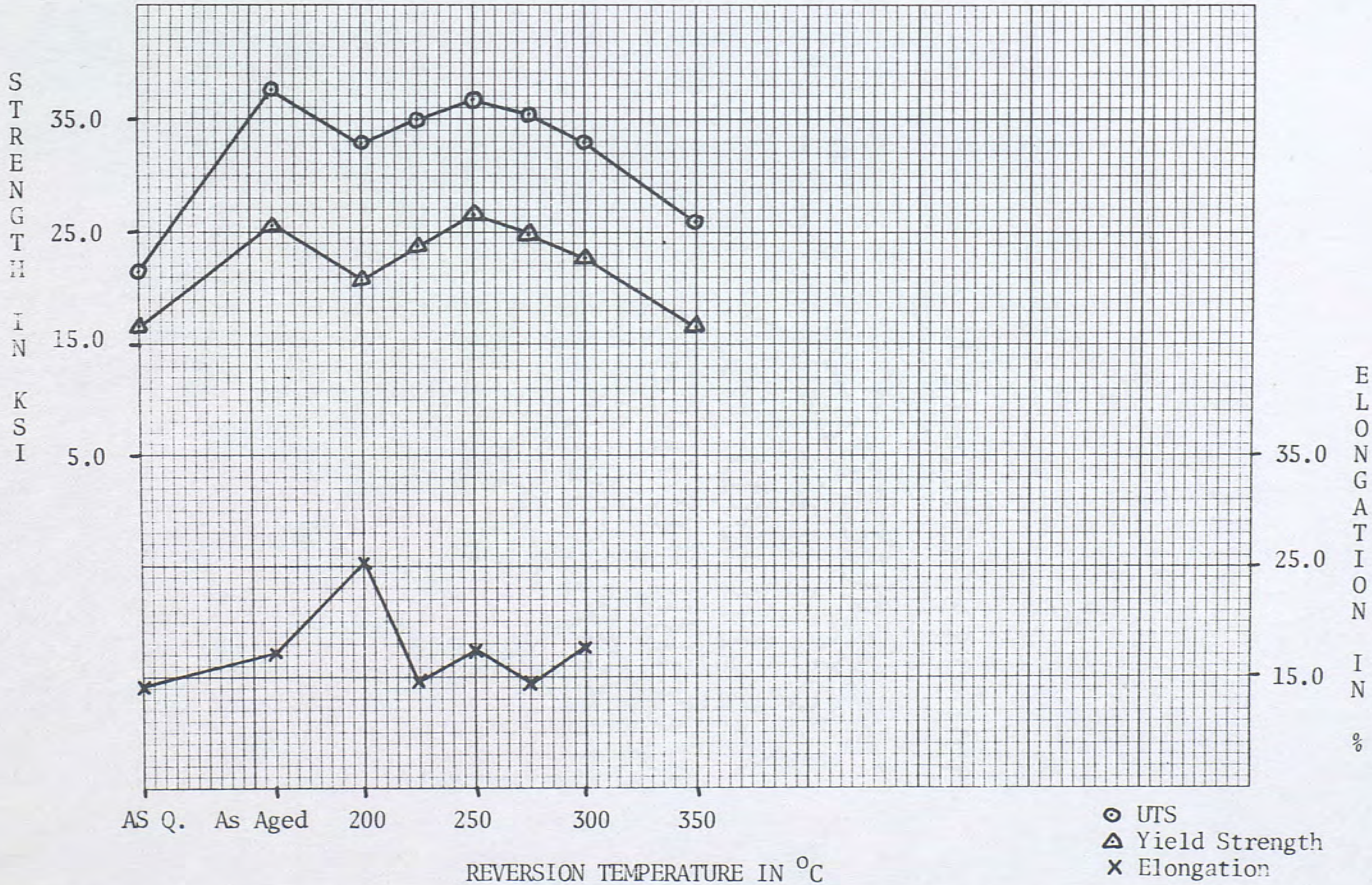

Fig. 11.--The effects of reversion treatments for 15 minutes at various temperatures on the tensile properties of a fully age-hardened Al-14 wt. % Ag alloy.

DISCUSSION

Single-Step Aging

The results obtained for the single-step aging experiments for both Al-Ag alloys follow the general trend observed in many other age-hardenable alloys. That is, in the case of the Al-14 % Ag alloy after 96 hours at 140^{o}C, the strength was steadily increasing. After 96 hours at 160^{o}C the age-hardening effect had leveled off and at 180^{o}C after 96 hours, over-aging was apparent with the rapid fall off in strength. These results are similar to those presented by Hansen [2], Köster and Braumann [5], Beton and Rollason [8], Davis and Nicholson [9], Yamaguchi and Ichimura [28], and Elliot and Axon [55].

The change in the rate of strengthening in the 1/2 to 8 hour aging time period is attributed to a phase transformation. Research by Auer and Gerold [24], Yamaguchi and Ichimura [28], and Baur and Gerold [52] indicates that in this region the η phase is transforming to γ'. The η phase is a type of G.P. zone state whereas the γ' is the intermediate metastable form of the equilibrium phase, γ (Ag_2Al).

Since the process of the nucleation of γ' on stacking faults [15,16], Frank sessile and helical dislocations [39,40], and at grain boundaries [40] is independent of the dissolution of the G.P. zones [39], such a change in slope would be expected [58]. That is, the two processes, one of the dissolution of the η- state G.P. zones and a second, that of the nucleation and growth of γ' could be taking place simultane-

ously [60]. In fact, it has been shown by Hren and Thomas [41] that the smaller G.P. zones dissolve in favor of the γ', supplying silver atoms for the growth of the metastable precipitate [9,41].

Even though the smaller G.P. zones near the γ' precipitate are dissolving [41], the volume fraction of the G.P. zones tends to remain the same during aging [23,27,52]. This is accomplished by the radius of the remaining G.P. zones increasing [20,28,52]. This would account for the increased rate of strengthening during the phase transformation of G.P. zones to the intermediate precipitate, γ'.

At long aging times the γ' precipitate is coarsening, as has been shown by Nicholson and Nutting [39], and Hren and Thomas [41]. Thus, the γ' precipitate becomes more incoherent with the matrix and the strength of the alloy decreases [37,39,41,43,58]. Further, the G.P. zones that are still present [39,41] become much larger than the critical zones radius and therefore, the contribution to the flow stress is greatly decreased.

After long aging times at 160°C, there is further indication that the γ' precipitate is still present because of the persistant increase in rate of strengthening [58]. This strengthening effect is associated with a decreasing difference between the UTS and the yield strength at long aging times. The tensile tests reveal values for the ultimate tensile strength, yield strength and elongation to fracture. Hardness tests, which were made by most observers who have investigated this alloy system, show only resistance to deformation in localized areas of samples and hence these measurements do not give representative bulk properties of the samples.

Two-Step Aging

In general, two-step aging did not increase or decrease the tensile strength properties of the Al-Ag alloys investigated. This is attributed to the similar size of the aluminum and silver atoms; the radius of the aluminum atom is 1.43 Å and the radius of the silver atom is 1.44 Å [62]. This leads to rapid diffusion rates of silver atoms in the aluminum matrix [9,12,17,18,32-35,37,41,49,52]. This is in contrast to the Al-Zn-Mg system which shows greatly increased strength properties upon two-step aging treatments [63]. On the other hand, the Al-Mg-Si alloys after many two-step aging treatments often show a decrease in the strength properties [64].

With extensive two-step aging treatments of the Al-14 % Ag alloy some strength and ductility was lost. For example, the aging treatment for one week at 80°C plus 24 hours at 160°C, the UTS was 2.0 ksi less than with the single-step aging treatment after 25 hours at 160°C. This loss in strength is probably due to the coarsening of γ' which becomes more incoherent.

Reversion Experiment

The reversion experiment results for the Al-14 % Ag alloy indicates that there are two reactions occuring simultaneously. One process is the dissolution of the ε- type of G.P. zones. The ε- zones are believed to be in a disordered state with a silver concentration of 30 at. % and are found upon aging above approximately 170°C [24]. The second reaction involves the growth of γ' platelets on stacking

faults [61]. At the lower reversion temperatures, i.e. 200^{o}C, the strength of the alloy is due to the G.P. zones and the γ' precipitate. A maximum in strength is obtained when the contribution of the two reactions reaches a maximum. Reversion above 250^{o}C leads to a decrease in strength due to the rapid coarsening of the structure [8,41].

G.P. zones are particularly stable in Al-Ag alloys [52]. They do not completely dissolve at a particular temperature, but instead they gradually dissolve while supplying silver to the γ' platelets [8,41]. Continued growth of γ' plates, leading to softening, which becomes pronounced after reverting at 250^{o}C, when the γ' precipitate becomes more incoherent with the matrix. However, G.P. zones and γ' precipitates, which remain after reversion treatments for 15 minutes at 350^{o}C, contribute some strengthening effect. This is shown by the UTS for this treatment still being higher than that obtained from quenching from the solution treatment temperature.

CONCLUSIONS

1. With single-step aging treatments, a phase transformation (from G.P. zones to the metastable precipitate, γ') is indicated by a change in the strengthening rate.

2. In most cases, the two-step aging treatments result in the same tensile properties as the single-step aging treatments for these Al-Ag alloys. This is attributed to the rapid diffusion of silver atoms through the aluminum matrix because of: (1) the lack of a size difference between the aluminum and silver atoms and (2) diffusion is increased because of the lower activation energy for diffusion of silver in aluminum.

3. In some cases, the two-step aging treatments lead to less ductile samples as is shown by the yield strength approaching the ultimate tensile strength.

4. With the alloys used, the single-step and two-step aging treatments did not show double maxima in the hardness vs. aging time curves as was shown by some previous researchers.

5. Reversion experiments indicate two reactions occurring simultaneously. These reactions are believed to be: (1) coarsening of the larger G.P. zones and the dissolution of the smaller G.P. zones and (2) the growth of the metastable precipitate, γ', on stacking faults.

6. Even after a reversion treatment of 15 minutes at 350°C, the strength of both the Al-6 wt. % Ag and the Al-14 wt. % Ag alloys is still above the strength of the samples quenched from solution heat treatment. This would indicate that either the remaining coarse G.P. zones and/or the coarse platalets of γ' are contributing to the strength of these alloys.

RECOMMENDATION FOR FURTHER STUDY

Further work needs to be conducted to definitely correlate the phase transformation with aging times and with aging temperatures. This would require the use of X-ray diffraction analysis and transmission electron microscope examination in conjunction with the tensile tests and aging treatments.

APPENDIX

TABLE 3

SINGLE-STEP AGING AT 160°C
Aluminum-6 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, age at 160°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
As Q	10.2	5.6	16.2
0.25 Hr	13.6	8.2	21.6
0.50 Hr	15.2	8.9	20.3
1 Hr	15.6	9.5	17.8
2 Hr	18.2	11.1	20.6
4 Hr	18.5	11.3	16.2
8 Hr	19.5	11.5	19.6
16 Hr	21.2	12.7	20.7
25 Hr	22.1	12.7	20.7
48 Hr	22.5	13.9	26.9
96 Hr	23.5	15.2	21.1
144 Hr	24.4	14.1	*
165 Hr	23.1	14.6	20.0

* Samples broke in the grips of the extensometer and no values were obtained.

TABLE 4

SINGLE-STEP AGING AT 180°C
Aluminum-6 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500° C for 20 minutes, quench at 20°C in water, age at 180°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
AS Q	10.2	5.6	16.2
0.5 Hr	17.7	8.9	23.4
1 Hr	17.6	9.6	21.8
2 Hr	17.0	8.6	20.5
4 Hr	18.3	8.9	21.7
8 Hr	19.5	9.9	18.4
16 Hr	20.1	10.6	18.1
24 Hr	20.4	10.9	23.1
48 Hr	20.1	11.3	18.8
96 Hr	21.4	13.2	17.8

TABLE 5

TWO-STEP AGING AT 80°C FOR ONE WEEK PLUS 160°C
Aluminum-6 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, 80°C for one week, then age at 160°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
1 Hr	15.2	6.5	16.1
2 Hr	18.2	9.2	19.3
4 Hr	19.8	9.8	13.8
8 Hr	21.6	12.3	20.8
16 Hr	21.6	12.7	21.7
24 Hr	22.3	13.0	19.8
48 Hr	23.3	12.7	22.0
96 Hr	24.0	12.9	20.0

TABLE 6

TWO-STEP AGING AT $100^{o}C$ FOR
24 HOURS PLUS $160^{o}C$
Aluminum-6 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at $500^{o}C$ for 20 minutes, quench at $20^{o}C$ in water, $100^{o}C$ for 24 hours, then age at $160^{o}C$.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
$100^{o}C$	12.9	7.3	33.1
4 Hr	19.4	11.1	*
8 Hr	20.0	11.5	15.8
16 Hr	20.7	12.2	*
24 Hr	21.7	12.1	17.7
48 Hr	22.7	13.3	21.8

* Samples broke in the grips of the extensometer and no values were obtained.

TABLE 7

REVERSION TREATMENTS
Aluminum-6 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, age at 160°C for 48 hours, quench at 20°C in water, then 15 minutes at 200°, 250°, 300°, or 350°C.

Aging Temperature	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
As Q	10.2	5.6	16.2
160°	22.5	13.9	26.9
200°	19.2	9.2	17.9
250°	16.9	10.3	20.0
300°	14.7	8.7	26.9
350°	10.8	4.6	37.5

TABLE 8

SINGLE-STEP AGING AT 140°C
Aluminum-14 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, age at 140°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
As Q	21.5	16.6	14.0
0.50 Hr	26.4	18.6	14.6
1 Hr	27.7	19.4	17.0
2 Hr	28.0	19.4	15.9
4 Hr	29.7	20.7	16.4
8 Hr	31.0	21.5	15.0
16 Hr	33.0	22.6	20.6
24 Hr	33.4	23.0	18.5
48 Hr	34.4	24.1	20.6
96 Hr	36.6	26.3	17.5
192 Hr	38.4	27.7	17.4

TABLE 9

SINGLE-STEP AGING AT 160°C
Aluminum-14 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, age at 160°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
As Q	21.5	16.6	14.0
0.25 Hr	26.3	17.8	15.0
0.50 Hr	27.2	17.3	12.1
1 Hr	28.8	17.6	16.0
2 Hr	31.6	19.7	15.0
4 Hr	31.8	20.6	*
8 Hr	32.2	22.3	18.2
16 Hr	34.8	24.1	19.1
25 Hr	36.8	25.0	20.5
48 Hr	37.6	25.5	17.1
96 Hr	35.7	28.2	10.3
120 Hr	37.6	29.1	*

* Samples broke in the grips of the extensometer and no values were obtained.

TABLE 10

SINGLE-STEP AGING AT 180°C
Aluminum-14 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, age at 180°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
As Q	21.5	16.6	14.0
1 Hr	27.4	15.4	20.5
2 Hr	28.9	17.3	19.0
4 Hr	30.6	18.2	16.6
8 Hr	31.4	19.7	19.3
16 Hr	33.2	22.4	15.1
24 Hr	36.9	25.2	11.8
48 Hr	36.8	26.5	9.8
96 Hr	34.5	25.8	*

* Samples broke in the grips of the extensometer and no values were obtained.

TABLE 11

TWO-STEP AGING AT 80°C FOR
ONE WEEK PLUS 160°C
Aluminum-14 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, 80°C for one week, then age at 160°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
80°	26.2	18.5	16.1
1 Hr	28.0	19.6	17.9
2 Hr	30.0	20.8	16.4
4 Hr	31.2	22.4	19.8
8 Hr	32.0	22.1	23.3
16 Hr	32.9	22.5	20.7
24 Hr	34.8	23.5	15.3
48 Hr	37.3	27.1	15.5
101.3 Hr	40.1	31.4	8.2

TABLE 12

TWO-STEP AGING AT 100°C FOR
24 HOURS PLUS 160°C
Aluminum-14 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, 100° for 24 hours, then age at 160°C.

Aging Time	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
100°C	24.6	18.1	*
4 Hr	32.0	22.2	16.6
8 Hr	33.8	21.4	19.6
16 Hr	35.2	24.8	18.3
24 Hr	35.3	24.8	17.0
48 Hr	36.5	26.5	*

* Samples broke in the grips of the extensometer and no values were obtained.

TABLE 13

REVERSION TREATMENTS
Aluminum-14 wt. % Silver

HEAT TREATMENT: Solid solution heat treatment at 500°C for 20 minutes, quench at 20°C in water, age at 160°C for 48 hours, quench at 20°C in water, then 15 minutes at 200°, 225°, 250°, 275°, 300°, or 350°C.

Aging Temper-ature	Ultimate Tensile Strength KSI	Yield Strength KSI	Elongation %
As Q	21.5	16.6	14.0
160°	37.6	25.5	17.1
200°	32.8	20.8	25.3
225°	35.0	23.8	14.5
250°	36.8	26.5	17.3
275°	35.4	24.9	14.2
300°	33.0	22.8	17.7
350°	26.0	16.7	*

* Samples broke in the grips of the extensometer and no values were obtained.

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