Effect of Ultrasonic Vibrations in Some Heat Treatments

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Abstract. The effect of ultrasonic vibrations on many heat treatment processes, primarily ageing, and others such as, hardening of solid and porous compacts, spheroidisation, etc. are described. Results obtained with regard to ageing are particularly promising and indicate the possibility of obtaining a distinctly shorter process time as also an increased ductility at the same strength. These features may be of interest to the aircraft industry.

1. Introduction

The industrial application of ultrasonic vibrations offers new possibilities¹ for reduction of process time or temperature or both as well as in an increase in mechanical properties of alloys through such treatment. The phenomena are probably caused by an increase in the number of lattice imperfections (dislocations, point defects, etc.,) which may bring about a faster reaction rate through aiding mechanisms like diffusion and precipitation. Diffusion is accelerated through the generation of point defects and the increase in strength is probably attributable to an increased density of strain embryos². During the last three decades work has been carried out on the use of ultrasonic waves as tools (for drilling etc)³. A number of publications pertaining to ultrasonic vibrations as an aid to heat treatment processes have, however, appeared only within the last decade, mostly from the Soviet Union.

Many investigators have drawn attention to the enhancement of diffusion of gases into solids in response to ultrasound. The ultrasonic treatment of steel in an ammonia atmosphere at 500°C has resulted in a significant improvement in the diffusion of nitrogen into steel and a similar effect has also been observed in gas carburizing¹. It has also been reported that the ageing process in some aluminium alloys is accelerated by 70-80 times in response to ultrasound. Low ultrasonic frequencies are found to be preferable for the production of finely dispersed structures and for accelerating the crystallization process in the solidification of ingots³. The structure of the ingot is improved as also its mechanical properties and deformability. The ingot shows more uniformity in properties although the gas content and chemical composition remain unchanged³. Palme⁵ found that duralumin aged in a vessel of water hardened more rapidly with ultrasonic vibrations passed through the water for a short period. In some cases vibrations not only accelerated room temperature and high temperature ageing but also led to an increase in the maximum hardness obtained although Jones⁶ reported little difference in the ageing behaviour of commercial aluminium alloys exposed to ultrasonic vibrations in a wide range of frequencies.

Bazelyuk, Polotskiy, Kashershaya, Sherman and Westerova⁷ found that the microhardness of AK 4-1 alloy — (an aluminium based alloy containing 5.5 per cent zinc, 1.9–2.5 per cent copper, 1.4–1.8 per cent magnesium) — increases with the exposure to ultrasonic vibrations after quenching. They also showed that the ultrasonic vibrations increase the strength and plasticity at high temperatures. There is an increase of 100 per cent in the high temperature plasticity. The time to rupture was found to be doubled and the steady state creep rate was reduced by a factor of four. Langenecker and Fauntain⁸ observed that ultrasonic vibrations having an intensity of the order of 25 watts/cm² at 20 kHz caused pronounced hardening of aluminium single crystals. An increase in temperature of the sample during ultrasonic treatment showed a decreasing effect on hardening. They also observed that slip traces never appeared on the surface of the samples indicating that the samples did not undergo visible plastic deformation during the ultrasonic treatment.

Bazelyuk *et al.*⁹ observed that, on exposure of polycrystalline aluminium to ultrasonic vibrations, dislocations multiply and point defects coagulate to form loops. On further irradiation the dislocations intersect with the Frank network. This substructure is found to be heat resistant leading to a significant reduction of creep rate. Bazelyuk *et al.*¹⁰ showed that as a result of exposure to ultrasonic vibrations there is a big increase in the resistance of copper specimens to high temperature creep. The time to rupture of copper specimens held under optimum conditions in an ultrasonic field was three times greater and the rate of steady state creep was 7.5 times less than for an unexposed specimen. There was also a considerable increase in hardness of copper on exposure. Unlike other treatments, exposure to ultrasonic vibrations appears to improve high temperature strength without affecting the geometrical dimensions of the specimens.

Vildanova *et al.*¹¹ found that the ductility of aluminium and of Al - Mg alloys increased when they were subjected to ultrasonic vibrations. Ramachandran and Dasarathy² observed that the final hardness of three steels, viz. a low carbon low chromium steel, a high carbon high chromium steel and an unstabilized 18-8 stainless steel increased when subjected to an ultrasonic bombardment.

Germanovich *et al.*¹² as well as Kuppuswami and Vasudevan¹³ and Bahl and Vasudevan¹⁴, have shown that infiltration of porous metals increases with ultrasonic vibrations.

When sound waves pass through a material they are absorbed¹⁵ due to a number of factors, the most important being the absorption due to imperfections in the lattice and the formation of point defects. According to Granato and Lücke¹⁶, the model hypothesized by Koehler¹⁷ of a pinned dislocation loop oscillating under the influence of an applied stress leads to two kinds of losses. One is the frequency dependent loss having a maximum in the megacycle frequency range and the other is strain amplitude dependent, in the kilocycle frequency range. Dislocations can be

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assumed to be pinned due to two causes : (a) interaction with point defects, and (b) interaction with a dislocation network. The latter is stronger. The dislocation loop length at first increases due to breakaway from pinning points and then decreases as dislocation multiplication increases the density of the network¹⁸. These changes are reflected in associated changes of velocity and attenuation.

A model of acoustic work hardening proposed by Langenecker¹⁹ assumes preferred absorption of acoustic energy at dislocation sites—perfect lattice areas are assumed not to attenuate sound noticeably. Due to large amplitude dislocation displacements, interaction with other dislocations may occur resulting in more defects and thus lead to an increase in the heights of potential barriers around the dislocations. In a subsequent tensile test, this would result in an increase in the static stress required for yielding of the specimen. This is called acoustic work hardening.

The most important physical effect of ultrasonic vibrations in liquids is due to cavitation. The results of cavitation may be spectacular and many ultrasonic effects are ascribed to the accompanying cavitation. Pressures are estimated to reach very high values²⁰ — even of the order of 10,000 atmospheres—when the ultrasonically generated cavities collapse and spherically propagating shock waves are produced as a result. By the resulting cascade process the shock waves spread themselves all around a specimen immersed in the fluid in a very short time and subject it to an intense bombardment. It is probable that due to this a part of the activation energy required for the precipitation process is given to the material. Thermal effects (due to transfer of sound energy to heat), although they become appreciable when frequencies go as high 300 kHz, are low at frequencies such as 25 kHz and the effect may therefore usually be neglected. Other effects are minor.

2. Choice of Problem

In view of the possible wide spheres of application of ultrasonics in Metallurgy and Heat Treatment in particular, it was decided to carry out some studies in the following main areas :

- (i) The effect of ultrasonic vibrations on ageing phenomena of 2024 and 7075 alloys (both *Al* based) when the waves are passed (a) through the specimens and (b) through the liquid medium in which the specimens are immersed.
- (ii) The effect of the vibrations on the quenching capability of a liquid medium in which the specimen is end quenched from the austenitic state.
- (iii) effect of vibrations on the quenching of sintered powder compacts
- (iv) effect of ultrasonic waves on the spheroidising operation
- (v) effect of ultrasonic waves on the tempering of hardened steel.

3. Experimental

(a) Equipment

The following items of equipment were employed to carry out the investigations :

- (i) An ultrasonic vibrator of IMECO type, 500 watts maximum capacity, 23 kHz, tank of dimension $15 \times 12 \times 10$ inches with lead zirconate titanate crystal.
- (ii) A locally fabricated variable frequency power supply, frequency range in 4 bands, 600 Hz to 2.6 kHz, 2 kHz to 8.3 kHz, 6.9 kHz to 22 kHz and 16 kHz to 47 kHz, power output 80 watts maxs (Fig. 1). The ultrasonic waves generated in a magnetostrictive material pass through the specimen mechanically coupled to it.
- (iii) A Hounsfield tensometer of 1 ton capacity, two strain gauges of 120Ω and 0.6 mm width fixed to one arm of a Wheatstone's bridge, a carrier frequency amplifier and an oscilloscope.
- (iv) A locally fabricated split die of heat treated hot die steel for pressing the powder compacts.

(b) Studies on ageing

The alloys 7075 and 2024 of the following compositions were taken; 7075 : 5.5% Zn, 2.5% Mg, 1.5% Cu, 0.3% Cr, rest aluminium. 2024 : 4.5% Cu, 1.15% Mg, 0.6% Mn, rest aluminium. Both were available as Alclad sheets. Cladding was removed by dissolution in NaOH. The 7075 alloy was solutionised at 490°C for four hours and water quenched. The 2024 alloy was solutionised also at 490°C (2 hr) and water quenched. The samples were aged at the following temperatures :

7075 : R. T. 80°C, 140C and 165°C

2024 : 120°C, 150°C, 175°C and 200°C

Some of the specimens were subjected to ultrasonic vibrations in the following fashion. Either they were given the treatment by immersion in oil agitated by ultrasonic waves for a flat period of 5 minutes (at R. T.) and then aged at the different temperatures as the alloy 7075; or they were immersed in the agitated oil for periods of 1, 2, 3 and 5 minutes *per hour* of ageing (at R.T.) and then aged at the different temperatures. The ageing treatment was given at 3 hour (ageing) intervals. In another series



Figure 1. Variable frequency power supply unit.

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of experiments, a similar set of 7075 samples were subjected to ultrasonic treatment by passing the waves through the specimen—(and *not* through the liquid medium)—using the equipment at (a-ii) at a fixed frequency of 23 kHz for 5 minutes. The hardness values are given as an average of several measurements. In addition, the U. T. S. and the percentage elongation were determined on the Hounsfield tensometer specimens (Fig. 2).

(c) Studies on hardenability

The materials used for this study were high carbon (1.1 per cent) steel and low alloy 4140 steel. End quench tests were carried out using simple cylindrical (non-standard) specimens of 12 mm dia and 75 mm length. The samples were heated to 770 and 860° C respectively and held there for about 90 to 100 minutes. The samples were hung vertically in the furnace using Kanthal wires. After the prescribed holding time, the samples were allowed to drop vertically with the bottom face just making contact with the surface of the ultrasonically agitated water. The hardness values were then measured on opposite flattened sides.

(d) Study on sintered compacts

Very fine turnings of 4140 alloy obtained from a local source were cleaned and ball milled to fine powder of less than 50 microns size. Non-metallic impurities were magnetically removed and the powder compacted in a locally fabricated die under a load of 7 tons into 10 mm dia specimens. Fine zinc stearate was used as the binder (< 1 wt per cent). The compacts were baked at 300°C to drive out the binder. The compact was sintered at 1100°C — (upto 700°C in hydrogen and thereafter in argon). After sintering, the compacts were quenched in water or oil, in both cases with and without ultrasonic vibration.

(e) Studies on spheroidising

Rod samples of high carbon (1.1 per cent) steels were first heated to 770° C and held there for 90 to 100 minutes before quenching in water. They were then taken to 650° C and held there before switching off the furnace. For the ultrasonic treatment, the rods after quenching were kept inside the magnetostrictive transducer. Care was taken to cool the transducer and the sample by dipping the transducer inside water. This is necessary as the specimens tend to get heated up quickly and if adequate precautions are not taken to keep the temperature of the specimen low, the effect of pure



Figure 2. Tensometer Specimen.

ultrasonic vibrations can not be isolated. The vibration was given for 5 minutes and then the samples were heated at 650°C and held for 6 and 8 hours respectively before cooling in the furnace. The resulting hardness is then compared with that of a specimen similarly treated except for the ultrasonic treatment.

(f) Studies on tempering of hardened steel

The material chosen for this study was medium carbon steel (0.5 percent C). The specimens were cylindrical ones of 11 mm dia and 75 mm length. The specimens were first soaked at 850°C for 1 hour before being water quenched to room temperature. The samples were divided into two sets, one set only was subjected to ultrasonic treatment for 3 minutes in a magnetostrictive fashion. (The Imeco agitator only was used but the output cable leading from the generator was not connected to the tank but wound over a cylindrical shell inside which the specimen was introduced. As there was a tendency for the specimen to get heated up very fast, the specimen together with the shell protected by a wax coating was kept immersed in water). Subsequent to ultrasonic treatment, the specimens were tempered at 100, 150, 250, 350, 450 and 550°C respectively—in all cases for one hour. The impact strengths of these specimens were then determined using an impact tester.

4. Results and Discussion

The results are given in Figs. 3-16.

(a) Effects on ageing of aluminium based alloys

In the 7075 alloy there is (Figs. 3 & 4) a sharp increase even in room temperature hardness after a five-minute ultrasonic treatment. A marginal increase in maximum hardness is seen in all cases although it is more evident at 140°C ageing than in other









cases (Fig. 5). The more interesting fact is the reduction in the ageing time for maximum hardness. Thus the time for peak hardness is reduced from 16 hours to 12 hours at 140°C and from about 30 hours to 20 hours at 80°C in the case of 7075 alloy. The most impressive observation of all is, of course, the effect at room temperature where a mere 5 minute ultrasonic treatment brings the same effect as a 28 hour ageing (without such treatment). There is however little difference in the peak hardness values, indicating that almost all effects possible at R. T. are obtained after 5 minute period and there is no further improvement thereafter. (Fig. 3). In the case of the 2024 alloy a similar reduction in ageing time has been noticed, viz. the time for maximum hardness reduces from 15 hours to 9 hours at 170°C after an ultrasonic treatment whose duration was worked out on the basis of 2 minutes/hour of ageing (Figs. 6 & 7). It is further noticed that there is no benefit in arbitrarily increasing the duration of ultrasonic treatment as can be seen from Figs. (10 & 11). The best results are obtained on 2-3 minutes treatment per hour of ageing, the effect declining thereafter. These results broadly agree with the results of the Russian workers. Figs. 8 & 9 show the variation of hardness with ageing time and temp. for untreated specimens. Figure 12 clearly shows that like hardness, the UTS and ductility are also improved on ultrasonic treatment. It appears as if a 5 minute treatment reduces the time to reach peak UTS at 140°C from 16 hours to less than 8 hours. Simultaneously the ductility seems to increase from about 9 per cent to 18 per cent. This is probably of very great significance in aviation from the fail-safe point of view. These results probably indicate that precipitation is not confined to grain boundary areas but is probably distributed all over the grain in a very uniform fashion. If so, it would follow that the corrosion resistance of the alloy would also be much improved





as one of the chief drawbacks of the duralumin alloys is their sensitivity to corrosion along grain boundaries because of selective precipitation in these areas. However no studies have so far been conducted by us with regard to the corrosion resistance aspect.

(b) Effect on hardenability

In the case of both water quenched and oil quenched specimens, ultrasonic vibrations produced increased hardenability (Figs. 13 & 14). Thus the distance from the quenched



Figure 8. Ageing temp. vs time to reach maximum hardness, duralumin 2024.



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% Elongation without U.S



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Figure 12. UTS and percentage elongation vs time, material 7075.

end at which the hardness reaches a value of R_c 50, increases from 23.2/16 to 33.5/16 of an inch in water and from 12.7/16 to 20.5/16 of an inch in oil. This is not surprising as the effect of even ordinary agitation in the quenching media is known for a long time²¹. The effect with ultrasonic vibrations is probably more marked. There was also a marginal increase in the hardness value obtained at the quenched end.

(c) Effect on sintered compacts of 4140 steel

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Because of the presence of pores, the hardness on quenching is not very high. Still the effect of ultrasonic vibrations passing through the quenching medium is to push up the hardness values. The values for quenching in water and oil are 221 and 139 VHN respectively. These go up, with ultrasonic agitation of the quenching medium,





Figure 16. Vibration time vs temp of specimen (°C) without cooling.

to 254 and 173 VHN respectively. It is possible that using more drastic quenches as 10 percent NaOH and ultrasonic vibrations of higher energy, one can considerably increase the hardness of quenched powder compacts. There is much scope for work in this area.

(d) Effect on spheroidising

Ultrasonic vibrations do not seem to bring about a quicker softening as one would *prima* facie be led to expect. In fact the results seem to indicate that the treatment seems

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to mitigate against too much softening. Thus the hardness values even after holding for 8 hours at 650°C is no less than VHN 286 (after ultrasonic treatment for 5 minutes at 90°C following water quenching from 770°C). The corresponding value for a specimen similarly treated except for the ultrasonic treatment is VHN 277. It appears as if ultrasonic waves, while they help dissociation of an unstable phase prevent any subsequent agglomeration of precipitated particles. Alternately one could consider that a great deal of fine nuclei are produced initially so that none could agglomerate to a great extent on subsequent holding at high temperatures. Further investigations are however going on to see whether this is truly so. It is to be expected that broadly similar effects are only to be expected even in the case of malleabilising of white cast iron.

(e) Effect on tempering of hardened steel

Impact values are distinctly improved in the range $100 - 350^{\circ}C$ (Fig. 15). After about $350^{\circ}C$ it was difficult to assess the benefits due to ultrasonic treatment as the specimens did not fracture after tempering at these temperatures even *without* such treatment. In order to assess the effect of the vibrations only, it is important to ensure that the specimen is not heated up during the treatment which can be very pronounced (Fig. 16). However, in real cases, cooling may not be necessary as one need not lose the advantage of the incidental heating due to the passage of the waves. The details of course would have to be worked out carefully first before embarking on the tempering operation.

5. Conclusion

The results of all the experiments detailed earlier clearly establish the advantage of using ultrasonic vibrations as a tool in different heat treatment operations. Probably any diffusion controlled process stands a very fair chance of being accelerated and in some cases even distinct improvements in the final mechanical properties can be achieved.

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