THIRTY YEARS OF RETROGRESSION AND RE-AGING

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Abstract

The first work on RRA was performed over thirty years ago by Cina and his colleagues in Israel, in an attempt to achieve high levels of both strength and stress-corrosion-cracking (SCC) resistance in 7075 aluminum alloy, thus combining the best features of the T6 and T73 tempers. Their process, however, involved such short heat-treatment times that its application would be effectively limited to very thin section components that typically do not exhibit SCC. Work at the NRC-IAR began in 1980, with the first goal being to explore modifications to the process so as to make it amenable to thick section parts. Since then extensive studies have been carried out on the kinetics, microstructure and structure-property relationships over a wide range of processing conditions and alloy compositions. This paper presents a review of the development of the RRA processes over the past thirty years, with an emphasis on the application of the technology to the aging aircraft problem.

1 Introduction

Despite the continuous development of new aluminum alloys and new variants of existing alloys, many aircraft parts and structures fabricated from 7xxx-T6 are prone to corrosion induced damage, including SCC and exfoliation. The issue has become increasingly important in view of the old age of many commercial and military fleets. Economical maintenance of these aging fleets is a major problem worldwide. A two-step heat treatment, known as Retrogression and Re-Aging (RRA), has been shown to give high corrosion resistance in 7xxx aluminum alloys equivalent to the T73 temper, together with the T6 strength levels. Therefore, the process is very promising as an effective means to control corrosion damage in aircraft components made using T6 material.

Since 1980, the IAR-NRC has been a major contributor to the development of the RRA technology. Work in this area has expanded on several fronts: a) real-time computer control, b) bulk processing of large and thick section (up to 4″x6″) extrusions and c) in-situ processing of aircraft parts made of 7075-T6511. Real-time process control is being developed and integrated with the heat treating equipment for process optimization, automation and quality assurance of depot level (or industrial) practices. Currently, the Department of National Defence, Canada, and the United States Air Force are both interested in employing the RRA technology for fleet maintenance or fuselage improvement programs. To these users, standardization of heat treatment and qualification procedures is important.

The authors feel that a historical review of the development of the RRA technology may be useful: we must learn from the past successes and even controversies and look forward to the new development and expansion of the RRA technology.

2 The Concept (1974-1984)

The concept of retrogression and re-aging (RRA) was first developed by Cina and his colleagues at the Israel Aircraft Industries in 1974 [1,2], which consists of two steps: 1) retrogression of the 7xxx-T6 material at an intermediate temperature between the aging temperature and the solutioning temperature, and 2) re-aging of the retrogressed alloy at 120°C for 24 hours. They experimented on 7075-T6, using a silicone oil bath, and they revealed that during retrogression the material's hardness/strength would first fall to a minimum before increasing again to a secondary peak, while continued treatment would cause a further decrease in strength. Thus there appeared to be three distinct stages to the retrogression process as illustrated in Figure 1. Cina et al. claimed that the optimum RRA treatment was to process the T6 material to the minimum strength, Figure 1, during retrogression, followed by re-aging using the original T6 aging treatment (24 hours @ 120°C). They developed the process as a means of achieving high levels of both strength and stress corrosion resistance in 7075 aluminum, combining the best features of the T6 and T73 tempers. Their process, however, involved such short retrogression times, typically 1 to 30 sec. at temperatures of 200-280°C, that its application would be limited to thin section parts to which SCC is not the critical failure mode, while 7075 aluminum is mainly used for thick section parts. Later, in an attempt to extend the treatment to larger blocks of 7075 aluminum alloy, Cina used induction heating to process some 3″ diameter cylinder blocks, but, because of the transient temperature gradient created by induction, the block could not be uniformly treated [3]. As a result, there was a shortfall in the corrosion resistance of the center material as compared to that of the surface material. Another limitation of the induction method would be to treat aircraft parts in asymmetrical shapes.

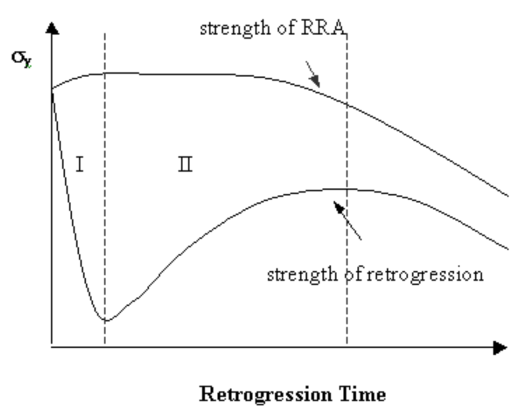

Figure 1. Schematic of the response of retrogression, retrogression and re-aging.

Realizing the limitations of Cina et al.'s original process, Wallace et al. in 1980 began to work on RRA, with the first goal being to explore modifications to the process so as to make it amendable to thick parts [4,5]. Using silicone oil as the heating medium and different processing temperatures ranging from 220°C down to 160°C, they found that the RRA benefit could be obtained under a wide range of temperature conditions. They claimed that the minimum retrogression point was not necessarily the optimum condition. They found that the process condition at the second retrogression hardening peak, Figure 1, where the material exhibits a higher electrical conductivity than at the "minimum" point, would give the material an improved stress corrosion cracking resistance, together with strength of T6 levels. Retrogression at these lower temperatures typically took 6 min. to 3 hrs., which allowed improved properties to be achieved through thicker section parts. Unlike Cina et al. who used unnotched coupons, they demonstrated the improved SCC resistance of the RRA material using double-cantilever-beam crack growth specimens (loaded in short transverse direction and immersed in 3.5% NaCl solution). A consistent trend of decreasing crack growth rate da/dt (the plateau velocity) with increasing electrical conductivity in aluminum alloys, as results of the long-time processing, was found.

Following the work of Cina and Wallace et al., many researchers conducted RRA research, using various heating media/methods. Kaneco at Lockheed used molten salt baths [6], Dubost and Bouvaist at Pechiney used a Wood's metal bath [7], as did Brown at Alcoa [8]. Tankins et al. at US Naval Air Development Center used molten salt baths [9]. Some rotary fatigue and bending testing of 7075-RRA material were performed in the early days as preliminary characterization of the structural properties of the RRA material [10].

These early studies demonstrated an intriguing combination of high strength and good corrosion resistance in essentially one aluminum alloy, which up to that point had been used primarily in two standard tempers, T6 and T73. The phenomena triggered a flourish of microstructural studies on 7075 aluminum alloy, trying to identify the controlling metallurgical factors. The first transmission electron microscopy (TEM) studies on the RRA material were conducted at NAE (former of NRC-IAR) [11,12], focusing on the microstructural changes and the mechanism of SCC retardation. Danh et al. [11] postulated that, during stage I, G-P zones present in the T6 condition were dissolving and the recovery of strength during stage II of retrogression was due to the precipitation of η′ precipitates and probably some reversion of G-P zones. Rajan et al. [12] observed the growth of grain boundary η precipitates in the RRA material, and following the argument first proposed by Christodoulou and Flower [13], proposed with supporting evidence that grain boundary η precipitates contributed to improved stress corrosion resistance by acting as the irreversible trapping sites for hydrogen. Accordingly, hydrogen produced by hydrolysis at the crack-tip was suggested to diffuse along grain boundaries until it meets an incoherent particle interface where it condenses to form small molecular gas bubbles, thereby lowering the hydrogen concentration in solid solution. This hydrogen trapping mechanism seems to suggest that the grain boundary precipitate size is a key factor of the material's SCC resistance. There are other proposed mechanisms which suggest that the corrosion resistance of aluminum alloys may be attributed to grain boundary inter-particle spacing [14-17], grain boundary precipitation-free zones [18,19], matrix precipitates [20,21], solute segregation [22-25], and dislocations [26,27]. These mechanisms are not totally contradictory, since most of these microstructural features are inter-related by the precipitation nature in commercial Al-Zn-Mg alloys.

Park and Ardell conducted a detailed TEM study on the microstructures of commercial 7075-T651 and 7075-RRA alloy [28,29]. They presented evidence of some precipitation phenomena, which were in conflict with the earlier findings that the T6 material contains predominantly G-P zones [30,31]. Park and Ardell found, instead, an abundance of η′ transition phase present in the commercial 7075-T651 plate. Hence, they concluded that the η′ precipitates should be the major strengthening agents in 7075-T6 material. They also observed that the RRA microstructure (e.g. retrogressed at 230°C for 30 seconds, and re-aged) also contained predominantly η′ precipitates and some η variants. Therefore, they proposed that the RRA processes involved first, dissolution of the finer η′ precipitates, and then, formation and coarsening of all the η variants. These processes would occur one stage later than Danh et al. had proposed (Danh et al. 1983), in the precipitation sequence of G-P zones→η′→η. It should be noted however that Park and Ardell used an electric spark discharge (ESD) technique to prepare their TEM samples in the form of 250μm thin strips. It was not clear whether the heat generated by ESD during the specimen preparation could have caused η′ precipitation to proceed such that the microstructures of their samples were affected.

3 Process Characterization (1985-1994)

During the second decade, intensive studies on the RRA microstructure(s) continued and extensive characterization and testing of the RRA materials were carried out [32-37], while the RRA processes were also being explored on many other 7xxx aluminum alloys. The RRA research also spread worldwide [38-53]. Alcoa filed a series of patents on processes similar to RRA [8,54-57], which were used primarily for 7X50 aluminum alloys and designated as T77 [58].

With regards to microstructure, Baldantoni [32] studied the differential scanning calorimetric (DSC) responses of three 7075-RRA microstructures (retrogressed at 220°C for 1, 4 and 6 minutes) in comparison with the T6 and

T73 microstructures. He observed that the first endothermic (dissolution) peak shifted from that close to the one for T6 to that for the T73 condition, indicating that the RRA microstructure contained more and more η′ with increasing retrogression time. The fact that the first endothermic peak of the 1 sec.- RRA microstructure falls within the temperature range above the G-P zone solvus, 150°C, and below the η′ solvus, 250°C [59], seems to support the postulation that G-P zones dissolution is the major precipitation activity during stage I of retrogression. Papazian also studied the microstructures of 7075 aluminum alloy in retrogressed-only and RRA conditions, using the DSC technique [33]. He observed a similar trend as the RRA microstructures changed from the condition processed at 220°C to that at 270°C (all for 60 sec.). For the retrogression-only microstructure (e.g., processed at 220°C), on the other hand, the first DSC response was an exothermic peak (precipitation) in the range of 120-160°C, which corresponds well with the G-P zone formation range. The above two DSC studies suggest that the RRA microstructure will change from the one that contains predominantly a matrix of unstable precipitates to one that contains more stable precipitates, either by increasing the retrogression time or by raising the retrogression temperature.

In an investigation on SCC resistance, Thompson at al. [34] compared the electrochemical responses of RRA material and its original 7075-T6 material and found that the cathodic current density was much lower in the former than that in the latter. Their results indicated that less hydrogen was produced in the RRA material than in the T6 material. The evidence supported Rajan et al.'s postulation that RRA material would contain less atomic hydrogen by allowing it to coalesce as hydrogen gas at the grain boundary trapping sites [12].

Tanlianker and Cina [35] also further investigated the SCC problem of 7000-type aluminum alloys, using TEM. They observed that more dislocations were present in the original T651 condition and the dislocation density decreased with retrogression, thus suggesting a clear relationship between dislocations present adjacent to the grain boundaries and the susceptibility to stress corrosion. Actually, Cina's original idea of RRA was in keeping with Jacobs's earlier findings that dislocations developed during quenching from the solution in the matrix or near the grain boundaries may be dissipated by heat exposure, and thus reducing the susceptibility of the material to SCC [26,27].

The above experiments and theories of RRA were reviewed by Wallace et al. [37] in 1990. In examination of Tanlianker and Cina's theory [35], Wallace et al. made a comparison of the microstructures of T6, T6RRA, and T73 materials (not cold-stretched and where dislocation densities were not high) with the corresponding T651, T651RRA, and T7351 materials. They observed little difference in the SCC behavior between T6 and T651, and between T73 and T7351 materials, in spite of differences in dislocation densities due to the cold stretching operations. Hence, they questioned whether dislocation density is the sole reason for the SCC susceptibility difference in 7000-type alloys. In the same study, they also presented S-L (loading in the short transverse direction vs. cracking in the longitudinal direction) fatigue crack growth data from the RRA material in comparison with the original 7075-T651 material. They found that the fatigue crack growth behaviors of the three materials were almost identical in argon, while in the 3.5%NaCl solution the RRA material exhibited slightly better fatigue crack growth resistance than the T651 material.

While most of the above studies were performed on small size coupons being processed under various isothermal conditions, some efforts were also spent on the treatment of thick-section blocks of 7xxx aluminum alloys [50,56]. The process for a thick section part would consist of first, an athermal heat-up process, and subsequently, isothermal soaking. At the time, no standard heat treatment profile or method was claimed to be universal for thick section parts, because the heat-up process would vary with the part size or geometry.

4 Application (1995-2001)

Since 1995, work at IAR has concentrated on RRA heat treatment of actual aircraft components made of 7075-T6511, e.g., C-130 sloping longeron. Holt et al. performed twenty-some RRA heat treatments on pieces of various size (up to 4′ long with maximum thickness of 0.75″) cut from a C-130 sloping longeron removed from service [60]. They explored various combinations of heating (autoclave/oil bath)/cooling (air/water/glycol) methods and media, and based on the results, they selected the profile of *40 minutes @ 195 ±2^{o}C* for retrogression treatment of the service-exposed parts. They achieved satisfactory strength in the RRA material, meeting the MIL-HDBK-5H A-Basis minimum requirements, and significantly improved the corrosion resistance (measured from ASTM EXCO exfoliation and C-Ring testing) of the material as compared to the original (service-exposed) 7075-T6511 condition [61,62]. The significance of this work was to expand the RRA process to actual aircraft parts, with varied section thickness, in service-exposed conditions. However, the process was defined empirically but not optimized from a microstructural point of view. The RRA heat treatments were then also performed on new 7075-T6511 extrusions of angle/channel sections and similar results were obtained as compared to the service-exposed material in the same temper [62,63]. Fatigue crack growth and fracture toughness tests were also conducted to demonstrate that there is no detrimental effect of RRA on the damage tolerance properties of the material.

The results of previous work have demonstrated the basic properties of 7075-RRA material, which are promising for structural applications. The next question would be whether this special heat treatment technology is applicable to large bulk material or in-situ on aircraft component? Since 1998, IAR has been concentrating on the RRA issues pertaining to depot level or industrial applications. The major issues are:

- When treated in furnace/media, what are the temperature profiles and their effects on large bulk materials?
- When applied locally to a component (in-situ), what are the effects of thermal gradients on the surrounding material, i.e. the heat-affected zone?
- What are the effects of RRA on top coat, primer and anodizing layers and vice versa?
- Can an optimized RRA condition be uniformly obtained regardless of the mass and shape of the parts, and whether being processed in bulk treatment or in-situ?

In the last three years, 1999-2001, under internal funding, IAR developed a precipitation kinetics model for the 7xxx series aluminum alloy [64] and successfully executed two RRA programs, one under contract with the United States Air Force/NCI on RRA of thick 7075-T7611 extrusions (up to 4″ thick) [65], and the other in collaboration with the Department of National Defence of Canada and the USAF/University of Dayton Research Institute on in-situ RRA of C-130 sloping longerons [66-68]. In both projects, the kinetics model was used to simulate the RRA condition under variable-temperature (athermal) conditions. Bulk treatment of 7075-T6511 blanks of 1″, 2″ and 4″ and 18″-long step beams were performed using an autoclave (as an air-circulating oven). Almost the same optimized RRA material condition, in terms of the electrical conductivity (~38.5%IACS) and strength loss (~ 3ksi), was achieved in blanks of all sizes, in spite of their variable thermal profiles. It should be emphasized that, for extrusions of thickness greater than 1″, only delta strength loss is the appropriate strength parameter for characterization of the RRA heat treatment, because the original material properties are not uniform, likely due to the uneven microstructure as a result of the extrusion deformation history. In-situ RRA was performed using small and distributed heater cells. Through this experience, plus the implementation of the RRA kinetics model in real time computer control, the technology has been developed to the point that it is applicable to aircraft components at the depot level.

5 Recent Results

Numerous strength and corrosion data have been published on RRA heat-treated 7075-T6 (or T651, T6511). In this paper, some results of the most recent work are presented, which are complementary to the existing body of information. The focus of RRA has been placed on 7075-T6511 recently, because the aircraft components that are prone to SCC damage are almost all extrusion materials [69].

TEM micrographs of 7075-T6511, 7075-T6511-RRA and 7075-T73511 conditions are shown in Figure 2. Since previous TEM work was mostly done on 7075 plates, these micrographs provide detailed microstructure information on the extrusions. Due to the limitations of the microscope, G-P zones cannot be seen in these photographs, but fine precipitates of η′ are present, especially in the RRA and T73511 materials. The amount of precipitation, including grain boundary precipitation, increases from T6 to RRA and to T73 condition. This general trend is in agreement with that observed in plate materials. Therefore, it would be reasonable to assume that the mechanism(s) responsible for reducing the SCC susceptibility of the extruded material would be the same as that in the plate materials.

The SCC susceptibility of thin (<0.75″) 7075 aluminum alloy extrusion has been characterized by Holt et al. [60,61], using C-ring specimens. In our recent work on thick (~4″) 7075-T6511 extrusions [65], the ASTM G-139 breaking load tests were conducted to quantify the material's short transverse SCC resistance in terms of the residual strength after corrosion. Figure 3 shows the residual strengths of 7075-T6511, RRA, and T73511, as function of time of immersion (in 3.5% NaCl) under different pre-stress conditions. It is seen that both the pure corrosion (at zero pre-stress) and SCC resistance of the T6511 material are very poor. The corrosion and SCC resistance of the RRA material is close to that of the T73511. Initially, the strength of the RRA material is significantly higher than the T73511 material. With increasing immersion time, the strength of the RRA material gradually decreases to values close to the T73511 material. This trend indicates that the RRA material may have a strength advantage over the T73511 material for a significant portion of the corrosion life of an Al 7075 component. On the other hand, the SCC life of 7075-T6511 material falls dramatically with even moderate pre-stresses (up to 60% of its yield stress). As another measure of the corrosion resistance of the material, ASTM G-34 EXCO ratings of 7075-T6511, T73511 and RRA conditions are given in Table 1. The above SCC and EXCO test results indicate that the maximum benefit of corrosion/SCC resistance may only be obtained with an electrical conductivity above 38%IACS. This, then, is our target for RRA heat treatment, but with optimization for minimum strength loss. The optimized RRA condition is quantified, in terms of precipitation fractions, by the kinetics model, and this condition is obtained in every RRA heat treatment using real-time computer control. Table 2 summaries the recent RRA results obtained at IAR with average strength levels all above the respective MIL-HDBK-5H A-basis allowables. The strengths of these RRA materials (with electrical conductivity > 38%IACS) are shown in Figure 4, with scatter bars indicating the variations of strength in these materials. The large standard deviation (~4.7 ksi.) in the 4″ thick extrusion, HA608, is due to the non-uniform microstructure of the material, whereas in other extrusions of net section thickness less than 0.78″, including service-exposed C-130 sloping longerons, the deviations are mostly less than 2 ksi. The last two in-situ treatments, performed with optimization by the kinetics model on HH02 and HK03, show a remarkable increase in the strength as compared with the results of the previous RRA treatments.

6 Conclusions

1. RRA, being a precipitation-controlled phenomenon, is applicable to 7075 aluminum alloys of all product forms (plate, extrusion, etc.), and may be extended to other alloys in the 7000 series.
2. The maximum benefit of SCC resistance can be obtained in Al 7075 with an electrical conductivity above 38%IACS, as demonstrated by the ASTM G-34 and G-139 tests.
3. For minimizing the strength loss that is inevitably associated with the RRA treatment, a kinetics model is needed to quantify the effects of RRA on microstructures of 7xxx aluminum alloys. These quantitative criteria can then be implemented in real-time computer control of the heat treatment. This is the most effective way for process optimization and quality assurance.

Acknowledgement

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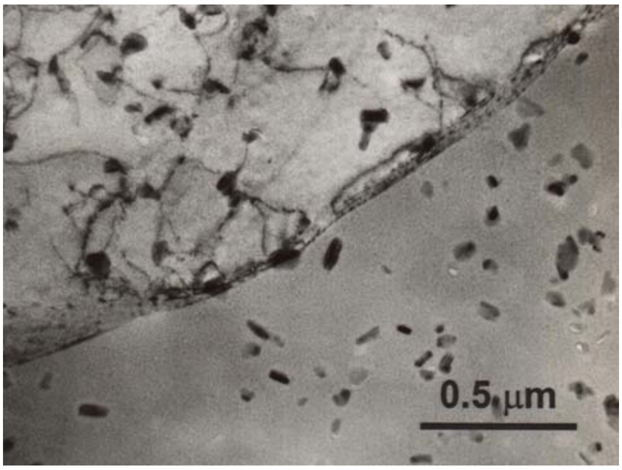
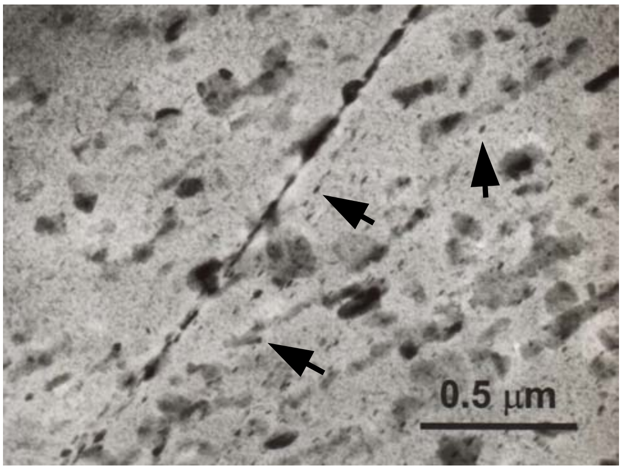
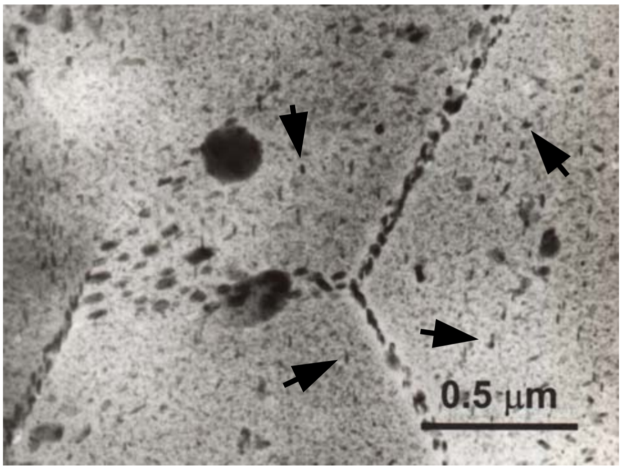


a) 7075 – T6511



b) 7075 – T6511 RRA



c) 7075 – T73511

Figure 2. TEM micrographs of 7075 aluminum alloy in a) T6511, b) RRA, and c) T73511 conditions. The arrows indicate η′ precipitates.

Table 1 – Exfoliation Results—ASTM G-34 Test

Material	Description	Hardness (HRB)	Conductivity (% IACS)	EXCO rating
Al 7075-T6511	As-received	89.5	32	EB/EC
Al 7075-RRA	Treated in autoclave	88.4	38.0	N/P
Al 7075-RRA	Treated in autoclave	86.4	38.2	P/EA
Al 7075-T73511	As-received	82.7	41	P/N
Al 7075-T73511	As-received	82.1	40.4	N/N

Table 2 – Strength of Different Al 7075 Extrusions by Different RRA Treatment

Heat Treatment	Material	Heat Treatment Details*	Date	Average Longitudinal YS [Ksi]		Average Longitudinal UTS [Ksi]	
				YS	Std. Dev	UTS	Std. Dev
First In-Situ	**Source E** Service Exposed C-130 Sloping Longeron Al 7075-T6511, 0.78 in Net Section Thickness (USAF Supplied)	In-Situ 195/40	July 1998	**72.2**	**2.0**	**80.8**	**1.8**
HA608	**Source A** New Al 7075-T6511 Extruded bar 4 in x 6 in (USAF supplied)	Bulk 195/10	July 2000	**72.0**	**4.7**	**81.3**	**3.6**
HE01	**Source E** Service Exposed C-130 Sloping Longeron Al 7075-T6511, 0.78 in Net Section Thickness (USAF Supplied)	In-Situ 195/37.5	March 2000	**71.8**	**3.0**	**81.1**	**2.1**
HH02	**Source H** Service Exposed C-130 Sloping Longeron Al 7075-T6511, 0.78 in Net Section Thickness (USAF Supplied)	In-Situ 195/34.8	Jan 2001	**77.1**	**1.6**	**84.5**	**1.3**
HK03	**Source K** New Al 7075-T6511 Extruded bar 4 in x 0.75 in (AMI Metals Inc.)	In-Situ 195/34.2	Feb 2001	**79.0**	**1.8**	**86.6**	**1.3**

*Heat Treatment details indicate RRA temperature and length of Retrogression Soak time. (I.e. 195/40 indicates a retrogression temperature of 195C for 40 minutes

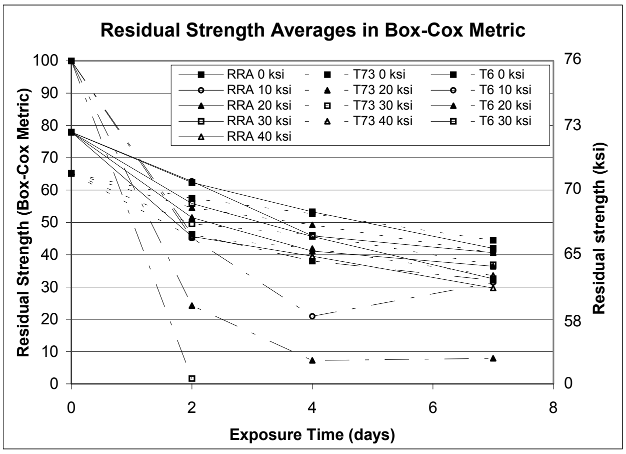


Figure 3. Comparison of residual strength, in Box-Cox metric, of 7075-T6511, RRA, and T73511 materials.

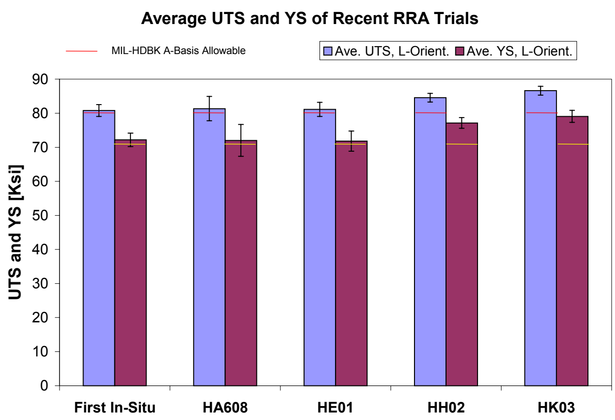


Figure 4. RRA Strength of Al 7075 extrusions obtained in recent years.

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