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TÍTOL DEL TFC: Characterization of the viscoelastic properties of aerospace aluminium alloys 2024 T3 and 7075 T6

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Resum

Sovint, el comportament viscoelàstic en metalls no es té en compte i és de principal importancia per certes aplicacions. És important, per tant, analitzar les propietats viscoelàstiques dels metalls i comprendre la física que les envolta per tal de predir correctament el comportament de metalls tant en aplicacions estructurals com en d'altres.

Els principals objectius són dos. Per una banda, es pretén obtenir mesures del mòdul elàstic mitjançant un procés experimental d'un seguit de provetes produïdes a partir dels aliatges d'al·lumini per aplicacions aeroespacials 2024 T3 i 7075 T6. Cadascuna d'aquestes provetes s'ha sotmès prèviament a un seguit de tractaments tèrmics. Aquests tractaments de referència es van extreure d'estudis previs documentats en articles científics. S'ha estudiat la influencia dels tractaments tèrmics en el mòdul elàstic.

Per altra banda, el segon objectiu era determinar les propietats viscoelàstiques de diferents provetes. Per relacionar les transformacions de fase i els canvis en l'estructura de l'aliatge d'alumini 7075 T6 amb els canvis observats en el comportament viscoelàstic s'han utilitzat anàlisis de tipus DSC.

El present document conté resultats i conclusions obtingudes un cop realitzats tant tests mecanico-estàtics com mecanico-dinàmics. Tots aquests tests es van realitzar amb un dynamic mechanical analyzer (DMA). Al ser aquest un instrument poc comú, la rara oportunitat de fer experiments s'entén com un valor afegit a aquest treball.

Els resultats obtinguts determinen que tant el mòdul elàstic com les propietats viscoelàstiques verien en funció del tractament de temperatura aplicat per cada proveta a més d'altres paràmetres com les freqüències de les càrregues aplicades al test dinàmic.

Title: Characterization of the viscoelastic properties of aerospace aluminium alloys 2024 T3 and 7075 T6

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Date: May, 28th 2009

Overview

Viscoelastic behaviour of metals is often obviated and is of paramount importance for some applications. It is important then to analyze the viscoelastic properties of metals, and to comprehend the underlying physics of this phenomena, so as to correctly predict the performance of metals for certain structural or non-structural applications.

The main objectives are two. On the one hand, a goal is to obtain measures of Elastic modulus by testing of several samples made originally of aerospace aluminium alloys 2024 T3 and 7075 T6. Each of the samples had been submitted previously to a different heat-treatments. These reference treatments were selected from previous works reported in scientific articles. The influence of the heat-treatments on the elastic modulus was studied.

On the other hand, the second goal was to determine the viscoelastic properties of the various samples. DSC analyses reported in literature have been used to correlate phase transformations and changes in microstructure of aluminium alloy 7075 T6's with changes observed in the viscoelastic behaviour.

Thus, the current document contains results and conclusions obtained by performing both static and dynamic mechanical tests. All these tests were done by means of a dynamic mechanical analyzer (DMA). The DMA is an un-common equipment. The rare opportunity of testing with it gives added value to this work.

The results obtained determine that the Elastic modulus as well as the viscoelastic properties vary depending on the applied heat treatments, and other test parameters like the frequencies of the applied forces, in the dynamic tests.

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INTRODUCTION

Low density materials not only have to be taken into account when designing aeronautic components but it is of paramount importance.

If we add strength and workability to lightness, we have three critical characteristics to consider in aerospace industry; aluminium combines and reflects them perfectly.

Metals' viscoelastic aspect is usually obviated or even not taken into account when analyzing its behaviour.

The objective of this document is not to expose an extended study of aluminium alloys, AA from now on the text. On the contrary, it intends to compare Elastic modulus and hardness values obtained by testing with literature and show the results obtained during the viscoelastic analysis done for AA 2024 T3 and AA 7075 T6, with no precedent, as long as the author knows. The rare opportunity of testing with a Dynamic Mechanical Analyzer or DMA enhances the value of the study.

To present this analysis, the document is divided into three parts. The first one presents both AA characteristics taking part in the study.

The second one explains the production process of the samples, presents the machines and tools used on it and describes both the methodology and the steps followed concerning the experiments.

Results and discussion will be attempted in part number three, as well as a comparison with results of previously-done studies.

Finally, the fourth chapter tries to collect the conclusions obtained.

"Within the course of the last two years ... a treasure has been divined, unearthed and brought to light ... what do you think of a metal as white as silver, as unalterable as gold, as easily melted as copper, as tough as iron, which is malleable, ductile, and with the singular quality of being lighter than glass? Such a metal does exist and that in considerable quantities on the surface of the globe."

Charles Dickens

1 CHAPTER 1. CHARACTERISTICS OF ALUMINIUM ALLOYS 2024 T3 AND 7075 T6

1.1 History

Both correctly named as *aluminum* or *aluminium*, this metal was officially established and named by Sir H. Davy in 1808. However, the first portion of this material is considered to be produced by H.C. Oersted in 1825.

The first aluminium companies in France, Switzerland and USA were founded in 1888. Famous authors as Charles Dickens or Jules Verne praised aluminium qualities as early as nineteenth century.

In 1903, the Wright brothers' first airplane had an engine modified with a 30-pound aluminium block in order to lower weight. Aluminium gradually replaced other materials as wood or steel, commonly used in airplane manufacturing by that time.

1.1.1 Aluminium metal production

Aluminium is produced with both primary aluminium and recycled aluminium. There are three basic steps to follow so as to produce aluminium metal:

- Aluminium ore or bauxite mining. Bauxite is generally extracted by open cast methods.

- Obtaining of alumina from bauxite. Usually using the Bayer process, which implies chemical and physical processes to finally obtain pure alumina.

- Obtaining of metallic aluminium from alumina. Applying a smelting process, alumina is electrolytically reduced to obtain metallic aluminium.

1.2 Basic Concepts

This section introduces the main magnitudes and theoretical knowledge related with the current study. It is divided into two parts: magnitudes appearing in the document and theoretical concepts.

1.2.1 Magnitudes appearing in the document

It is worth to explain the most important magnitudes related with the viscoelastic analysis. These are presented after these lines.

Elastic or Young's modulus (*E*)

The Elastic or Young's modulus is a mechanical property of materials which provides information about the elastic behaviour of a material as well as its strength. It is related to the stiffness of materials: the capacity to resist elastic (reversible) deformations. Diamond has one of the highest known elastic modulus (*E*=1.000 GPa). For a linear elastic and isotropic sample to which we apply either compression or traction load, *E* will remain constant. Hooke's Law applies in this latter case (see below). Conversely, non-linear elastic structures cause the *E* to vary.

$$E = \frac{\sigma}{\varepsilon} \quad \textbf{(1.1)}$$

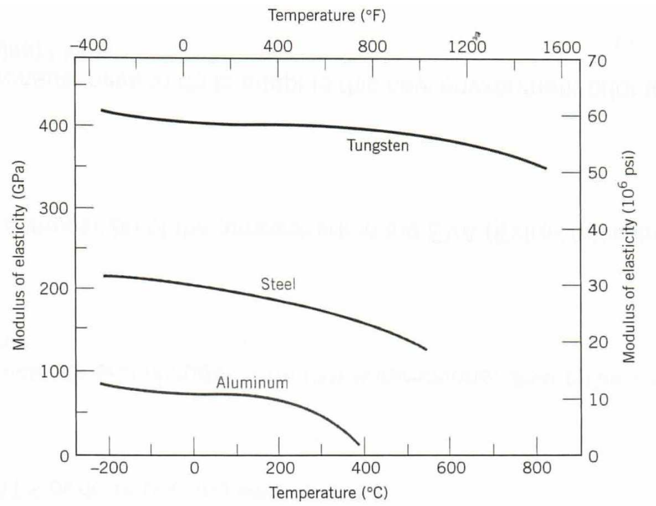

Figure 1.1 Elastic modulus variation with temperature for several metals: tungsten, steel and aluminium. [1]

Stress (σ)

Stress is defined as a force per unit area that acts in a way which tends to change the shape of an object (tends to cause deformation). This magnitude is measured in [Pa].

Strain (ε)

The deformation experienced in a material after being submitted to a force is known as strain. It is a non-dimensional magnitude and is commonly measured in [%] or [microstrains].

Hardness (HV)

Hardness is another mechanical property, referred as the capacity of a material to resist plastic deformation locally or in its surface. Typical values of hardness for AA 7075 T6 and 2024 T3 are HV160 and HV120, respectively. The hardness test as well as its implementation method will be explained in-depth later on. All hardness tests performed by the author were standardized Vickers Hardness tests.

$$HV = 0.1891\left(\frac{F}{d^2}\right) \quad \textbf{(1.2)}$$

Storage and Loss moduli

Viscoelastic behaviour of a material can be described by its storage and Loss moduli, also defined as *E'* and *E"*. [2]

The first provides information about the stored deformation energy, linked with an elastic behaviour and the other allows us to estimate the amount of energy lost as heat which is closely related to a viscous response.

$$E' = \left(\frac{\sigma_0}{\epsilon_0}\right)\cos\delta$$

and

$$E'' = \left(\frac{\sigma_0}{\epsilon_0}\right)\sin\delta, \quad \textbf{(1.3)}$$

The phase of the expressions above corresponds to the lag between stress and strain signals once applying a load to the sample. To compute these values, the FS-TS is done **(1.4)**.

$$\epsilon = \epsilon_0 \sin \omega t,$$
$$\sigma = \sigma_0 \sin(\omega t + \delta)\,. \quad \textbf{(1.4)}$$

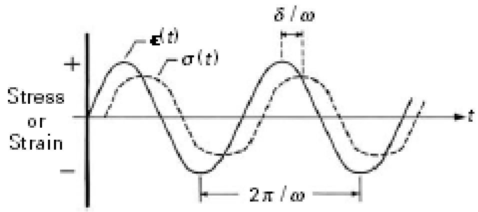

Figure 1.2 Stress and strain signals. The DMA studies time lag between these signals to describe the material's viscoelastic behaviour. [2]

1.2.2 Theoretical concepts

Elasticity

Elasticity is defined as the capacity of a material to recover the state it had before being stressed once the force applying on it is released.

Elasticity is defined as the capacity of a material to recover the state it had before being stressed once the force applying on it is released.

A linear elastic material maintains constant its Young's modulus or elastic modulus (*E*) when applying a load on it. If we think about this concept and take into account formula 0.1, we can rapidly observe what the latter expression means: the stress vs. strain plot in the elastic region will be represented as a straight line (slope is constant and equal to E). Concerning pure aluminium, the characteristic *E* is about 72,2 GPa.

However, the elastic response of a material may be also non-linear (see stress-strain plot in **Figure 1.3**). In this case, although the material's elastic behaviour is not linear, upon release of the load, the sample would also return to its initial shape, and no permanent deformation would be observed.

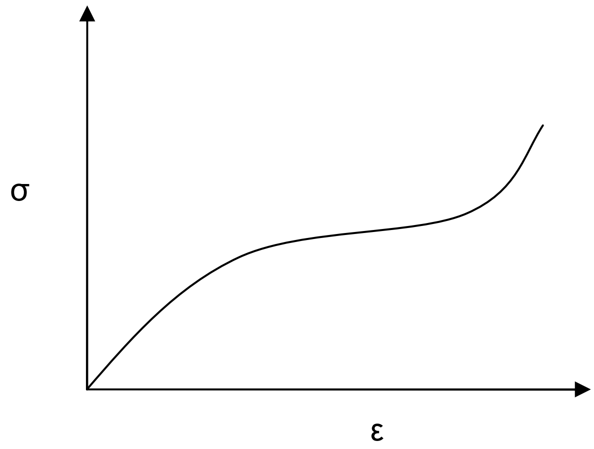

Figure 1.3 Stress-strain plot for a rubber.

Plasticity

On the other hand, we define plasticity as the capacity of a material to deform plastically when stressed. Plastic deformations are permanent (non-reversible nor recoverable)

Most of the materials, as in the case of aluminium, present an elastic and a plastic zone in their stress-strain diagram, which characterizes their response to stresses. By convention they are separated by a point called yield stress. It is important to point out that most of the materials we know today do not present a pure elastic or pure plastic behaviour. They mainly present both an elastic and a plastic region when submitting them to a Stress-Strain test.

Viscoelasticity

Probably viscoelasticity is the most important concept to understand as it is the necessary basis for this work.

We define viscoelasticity as the property of a material which present both an elastic and a viscous behaviour.

The ideal solid behaviour do not imply viscosity when not submitted to high temperatures. As we increase this temperature up to high values, the original crystalline structure of metals begins to diffuse atoms or molecules within the material. This flow of molecules taking place in the microstructure causes viscosity to appear; from this point, the metal we are dealing with does not behave as a pure metal: it behaves as both a solid and a fluid.

In order to characterize the viscoelastic behaviour of a material, it is sinusoidally deformed while its stress values are recorded. An ideally elastic element would return to the original state the material was before applying a deformation at the same time the force that causes it releases. When a time lag between the material response and the release of the force once deformed exists a phenomenon called viscoelasticity appears. [2]

This time lag (δ) is computed as a parameter and is used to compute the Storage and Loss moduli, as explained above.

Processes applied to the samples

Ageing

The ageing process consists on submitting the samples to an specific temperature for long periods of time. Doing this, the microstructure organizes itself to achieve a metastable state –which means that this stability is temporary, following processes will again disturb lattice's equilibrium-. It can be made at room or at an artificial temperature. Mainly, this treatment is applied to improve specific strength properties.

Average time required to apply this process is about 24h.

In this document, ageing refers to a heat treatment applied before retrogression procedure. It is applied by the metal supplier as it takes part of the T3 or T6 treatment (in-depth explained further).

Retrogression

This process modifies several specific properties of the microstructure depending on the parameters temperature-time we choose. It is probably one of the key processes to take into account in a strengthening procedure.

Average time to perform this process goes from seconds to three or four hours.

Retrogression is referred as the heat treatment applied after ageing. All heat processes include a retrogression step.

Re-ageing

A new ageing is applied after retrogression to restore an equilibrium in the lattice. It is worth to mention that to perform this study it has been applied artificially to all samples (including AA 2024 T3); however, once the sample has suffered all heat treatments required it also suffers a room temperature ageing while being stored so this must be taken into account. However, this process is not considered when analyzing the results (explained further).

Average time required to apply this process is, as ageing, about 24h.

The re-ageing process is understood as the last transition treatment before testing. As ageing and retrogression, re-ageing takes place in all heat processes.

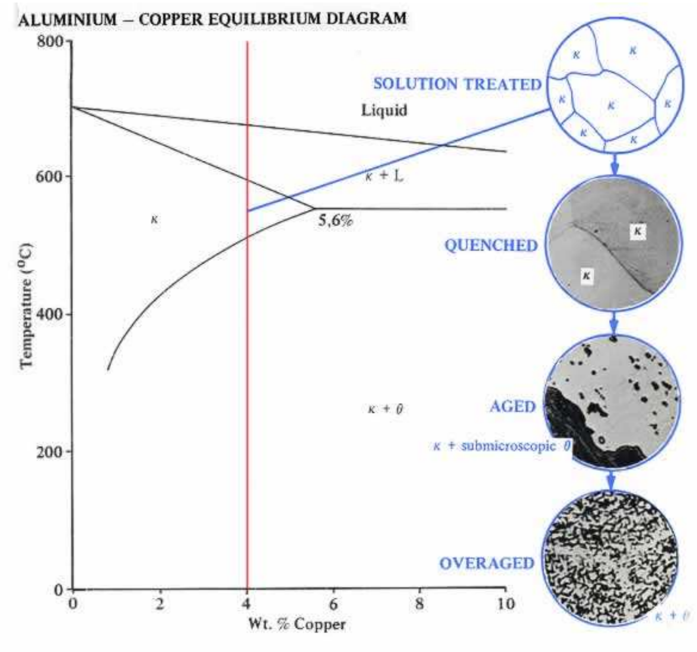

Figure 1.4 Changes in microstructure for AA 2xxx series. © State of New South Wales through the Department of Education and Training and Charles Sturt University

Table 1.1 Reference values of temperature and time for different heat treatments applied to AA 2024 T3 and AA 7075 T6.

Heat treatment	Refference T (ºC)	Refference time
Ageing, Re-ageing	120-130	24-38 h
Retrogression	160-220	0,5min-250min

1.2.3 Evolution of the microstructure

The objective is to obtain a matrix of solvent and solute in equilibrium state. To reach this point, the AA has to overcome a couple of states characterized by different elements explained in this section:

SSS → GP zones → η’ → η → matrix in equilibrium

As a matter of fact, the process presented above these lines is exclusive for AA 7075 T6. The process for 2024 T3 does not include *η’ or η* but θ’ and θ. These parameters refer to different composition elements which suffer a couple of changes along its corresponding process. To explain these steps, just the series shown above is considered.

1.2.3.1 Changes in AA matrix

The first step is to dissolve all the alloying elements at a high temperature. Once this process is finished, the plate is quenched obtaining higher values of nominal hardness than the solvent metal when in pure metal form. A supersaturated solid solution (SSS) is formed, as it contains more solute than in its solubility equilibrium level. After this process, vacancies and solute atoms become frozen and thus this saturation state lends the new AA a microstructural matrix full of vacancies and dislocations created by the excess of solute in the microstructure. Until now, the grain boundary structure has not been affected. This process is made by the aluminium alloys supplier.

If we apply an ageing heat treatment on the supersaturated solution, the lattice experiences some different stages due to temperature changes.

As the temperature increases, different areas where the solute creates a coherent structure in the SSS matrix appear. These are called *Guinier-Preston zones, or GP zones* taking its name from the two researchers who first discovered them. Concerning its nature, these areas are very small and for this reason they do not affect the lattice deeply.

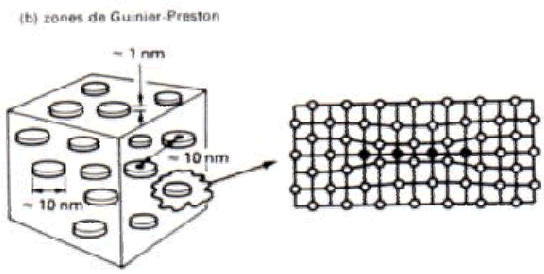

Figure 1.5 *GP zones*. [3]

As well as the temperature increases, these new coherent structures involve also new strains in the atomic structure. Semicoherent zones (also known as η') begin to appear and with them, dislocations created by the matrix in order to take up strain forces. In **Figure 1.4** we can see different states for the matrix. The first one shows a complete coherence between solute and solvent's matrix; the second one presents still a coherence but with a strain. In the third figure, there is a partial continuity of lattice rows across the interface and the last one represents incoherent equilibrium precipitate as interface or solute's matrix and solvent's are completely unrelated.

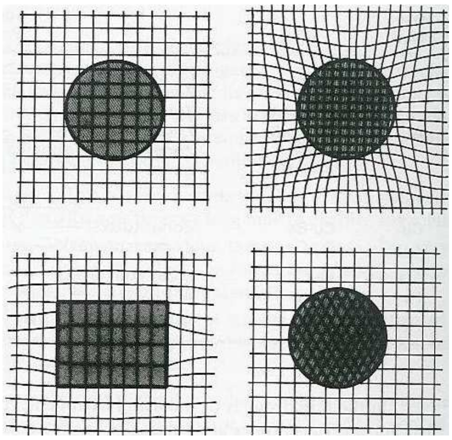

Figure 1.6 Crystallographic relationships between precipitate lattice and the solvent matrix. [2]

As a result of further and continuous growth of these η', the matrix and the precipitate appear divided by an incoherent interface where the lattice elements begin to positioning themselves in a smoother manner: again, coherent structures (η) appear and create a structure having higher strength. However, this temperature increase can not continue indefinitely; the reason is that not only these η' and η zones grow, but also the grains. This is the reason why the heat treatments applied to an AA must be so well studied for each composition. A compromise between temperature, strength/hardness and time value for each treatment is required.

1.3 Properties of Aluminium Alloys

The current part describes important properties of the studied aluminium alloys. In **Table 1.2** a comparison between properties of pure Al and those of the studied aluminium alloys is presented. [4]

Table 1.2 Main properties comparison between pure aluminium and aluminium alloys employed.

	Structure	Tensile Stress [MPa]	Yield Stress [MPa][1]	Max. elongation [%]	Density [g/cm^3]	Fusion temperature [ºC]	Young's modulus [GPa]
Pure aluminium	FCC	440	325		2,7	660	72,2
2024 T3	-	435	290	12			
7075 T6	-	525	460	6			

Aluminium is malleable and if we compare steel's and titanium's density, 7.8 g/cm^3 and 4.5 g/cm^3 respectively, with aluminium's 2.7 g/ cm^3, we clearly notice its extraordinaire lightness, which leads to good specific mechanical properties. These are probably the most critical properties for an aerospace structural material. Moreover, durability and good behaviour against fatigue and cracking phenomena are also considered important parameters to deal with in structural design of aircraft and other aerospace vehicles.

However, aluminium presents relatively bad values of strength and a low fusion temperature. Actually, in terms of hardness, elastic or Young's modulus and ultimate tensile strength, aluminium hardening is required for structural applications. To do so, three main methods can be employed:

- Solid solution strengthening.
- Cold work hardening strengthening.

[1] Calculated stress for a 0.2 % elongation.

- Precipitation strengthening.

All of these three hardening methods can be applied to Al. It is important to point out that strengthening using cold work hardening can be applied to Al as its structure (FCC) favours easy movement of dislocations along its slip systems. Nevertheless, as the level of deformation of the material increases, further cold work causes lower variation in hardness values, up to saturation or breaking of the piece.

A good way to achieve also improved mechanical properties is by causing precipitation. The difference between this method and solid solution lies in temperature applied to each one of them. Higher values of temperature also increase solubility and this favours some elements to experience solubility enough to become part of the solution.

In this latter method there are four important points to take into account when proceeding:

- Talking about the solution process, the temperature to which our alloy is submitted must be adequate, as well as the time of exposition at the given temperature.

- Once this is accomplished, the next step is to choose the best cooling method (quenching, low temperature reduction, etc.).

- In terms of ageing, this can be natural which implies temperature values between 20 and 30 ºC or artificial that usually involves much higher temperature levels. These processes stabilizes the microstructural matrix and is often applied after a treatment that implies high temperatures. See chapter 1.

- Once an AA is produced, different processes to improve mechanical behaviour are used depending on the final application of the corresponding piece. Focusing on this document, AA 2024 T3 and AA 7075 T6 have been chosen.

1.3.1 Nomenclature of aluminium alloys

The numeric series indicate to which family of alloys is associated the particular AA (which depends on the relative importance of alloying metals) and the following letters and numbers refer to the specific treatment applied to improve the AA properties. These processes are standardized.

Letter T is used to define heat treatments for structural strengthening. These treatments can include cold work hardening process or not and are always followed by a number that defines the specific sequence followed for each treatment.

Table 1.3 Sequence of treatments.

T3	Solution, quenching, cold work hardening and natural ageing
T6	Solution, quenching and artificial ageing.

To learn more about definitions of Al state treatments read [5].

1.3.2 AA 2024 T3 and AA 7075 T6

Aluminium alloys AA 2024 T3 and AA 7075 T6 have been chosen because they are both extensively employed in aircraft structures owing to good response to fatigue phenomenon for the first one and to high values of mechanical properties after age hardening for the other.

The main elements present in AA 2024 are Cu, Mg and Mn, in decreasing order. AA 2024 has much lower resistance to corrosion. However, it has good fatigue properties, resistance to cracking propagation and fracture.

Practical applications for AA 2024 T3 correspond to areas or zones where the aluminium structure or piece is submitted to high fatigue and deformation strains and a good behaviour against them is required.

The AA 7075 T6 overhangs 2024 T3 in terms of strength properties (ultimate tensile strength and yield stress). For this reason, it is usually used as structural material in areas where high compressions and traction forces are feasible.

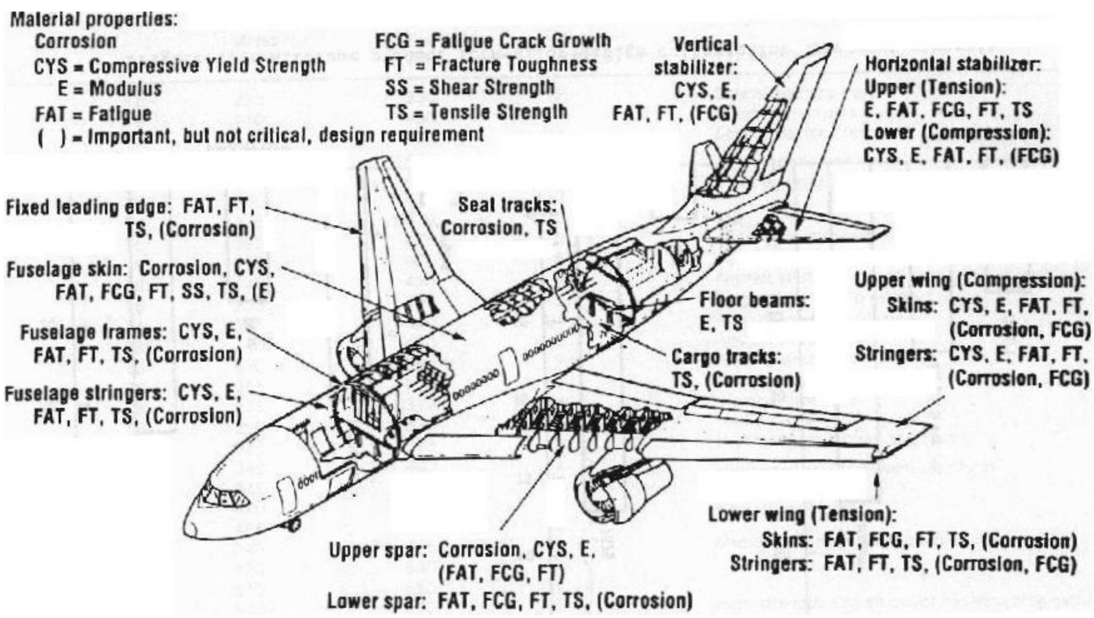

Figure 1.7 Requirements for the components of an aircraft. [6]

1.4 Current applications in aeronautics

One of the most important applications for both AA 2024 T3 and 7075 T6 in aeronautics is as aircraft structural materials, due to their high strength and low density values. **Figure 1.8** shows a sketch of an aircraft and the materials that are usually used for each corresponding structural part.

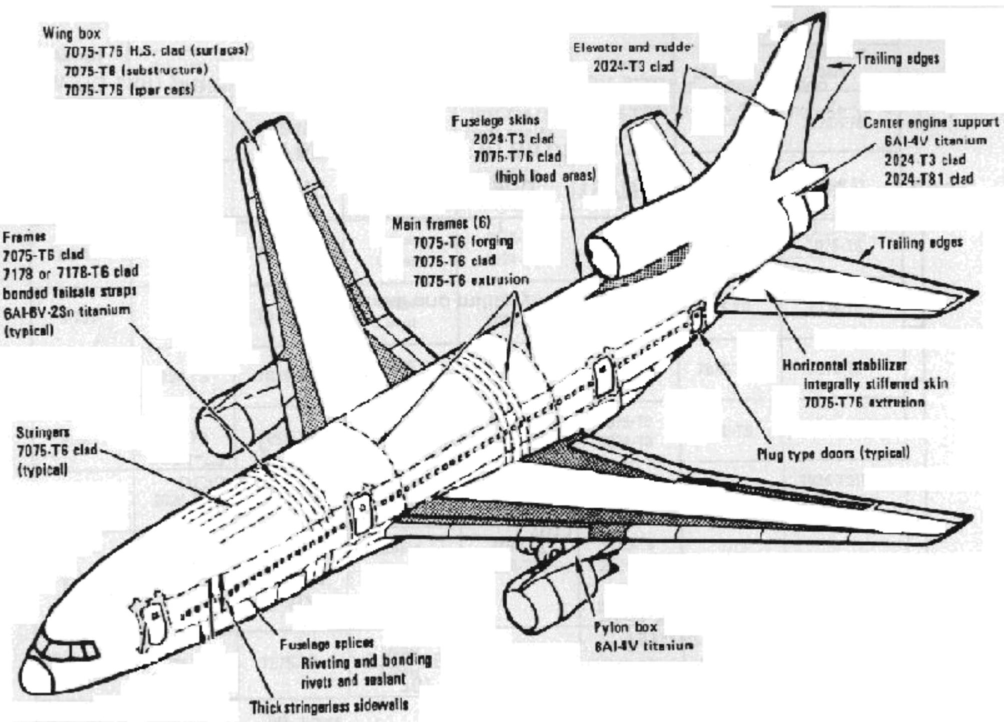

Figure 1.8 Materials used in the DC-10 aircraft structure. [6]

In aeronautics, AA 2024 T3 is utilized as fuselage's skin panels, in the wings' lower surface and other parts where fatigue is a critical parameter of design.

AA 7075-T6 is used in wing's upper surface and is widely used in fuselage and other zones where high resistance and hardness play a crucial role.

Besides aircraft structure, AA 2024 T3 is commonly used in so diverse fields as manufacture of truck wheels, scientific instruments, veterinary and orthopaedic paces. Lacrosse shafts and mould tools are examples of elements that employ AA 7075 T6.

2 CHAPTER 2. EXPERIMENTAL

2.1 The Samples

The samples consist on rectangular-shaped laminae having 60 mm length, 15 mm width and 2 mm of thickness. Some samples were 10 mm in width, but as expected, this fact seems not to influence the results. These pieces are cut from two different aluminium alloy mother-plates: one of AA 2024 T3 and another of AA 7075 T6 with a chemical composition as indicated in **Table 2.1** and **Table 2.2**.

Table 2.1 Chemical composition in weight [%] of AA 2024 T3 samples.

Si	Fe	Cu	Mn	Mg	Zn	Ti	Cr
0,180	0,280	4,460	0,640	1,350	0,040	0,050	0,010

Table 2.2 Chemical composition in weight [%] of AA 7075 T6 samples.

Si	Fe	Cu	Mn	Mg	Zn	Ti	Cr
0,060	0,150	1,500	0,010	2,580	6,000	0,050	0,190

Once the samples were machine cut, each one of them was submitted to a certain heat treatment so as to perform the study. These processes are explained in-depth later in this chapter.

In **Figure 2.1** you can see a detail of the samples used.

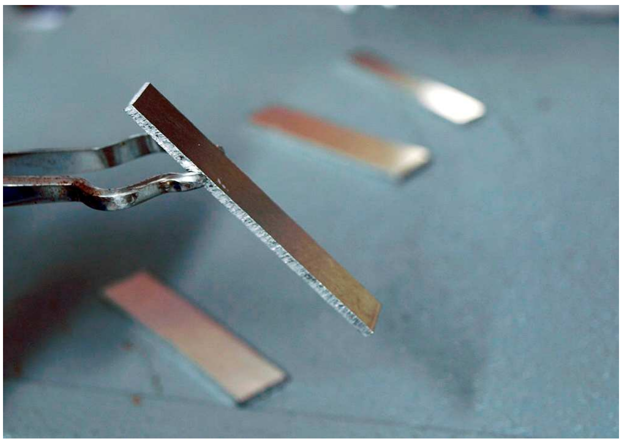

Figure 2.1 Samples detail.

2.1.1 Choice of Treatments

Heat treatment processes allow increasing hardness and strength values of aluminium alloys, as in other materials; but can also improve fatigue behaviour and other properties (corrosion, electrical conductivity) of these AA. Later in this chapter, these different procedures are attempted.

From now on, we will refer to the whole set of heat treatments as heat process. This is, for instance, we will refer to an ageing as a heat treatment and the whole set of heat treatments applied to a sample (namely, ageing, retrogression and re-ageing) will be referred as a heat process.

The treatments applied to the samples have been selected from scientific articles and their selection has been ruled by the following aspects in order of priority:

- Its relation with viscoelastic properties.
- The amount and quality of data presented.
- Whether they deal with AA 2024 T3, 7075 T6 or both.
- Methods of analysis.
- Other: including interesting theory, aluminium production processes or aluminium recycling methods.

Table 2.3 Origin of treatments.

Extracted from	Heat process
[7]	R at 160ºC for 29 min and RA at 120ºC for 38h
	R at 180ºC for 9 min and RA at 120ºC for 38h
	R at 200ºC for 2 min and RA at 120ºC for 24h
	R at 220ºC for 0,5 min and RA at 120ºC for 24h
[8]	R at 180ºC for 40 min and RA at 130ºC for 12h
	R at 200ºC for 30 min and RA at 130ºC for 12h
[9]	R at 200ºC for 5 min and RA at 120ºC for 24h

Each one of these treatments has been applied to both AAs. This implies a total number of 14 different samples plus 2 without heat treatment or as-received, one per each type of aluminium alloy.

2.2 Dynamic Mechanical Analysis

In this section the main properties and characteristics of the Dynamic Mechanical Analyzer (DMA) used to perform the Dynamic Mechanical Analysis tests as well as its model equations are described.

2.2.1 Dynamic Mechanical Analyzer (DMA)

A DMA is a precision instrument used to measure mechanical and viscoelastic properties both on rigid and soft solid materials, under controlled temperature settings. Concretely, storage modulus and loss modulus together with temperature are key parameters in the Frequency Sweep-Temperature Step test (better explained later in this chapter) while stress and strain parameters characterize the Stress-Strain test, also detailed further on.

The samples are mounted on a clamp with a stationary and a movable part connected to the drive motor. Thus, the motor affects directly the deformation of the sample. The way it works is simple: the drive motor delivers force or stress to the tested sample while an optical encoder measures the resulting displacement. Furthermore, it includes an air bearing of nitrogen to assure a smooth and continuous delivery of force without noise.

The instrument used to perform Dynamic Mechanical Analysis tests on our AA samples is the *TA Q800 DMA*. See **Figure 2.2**.

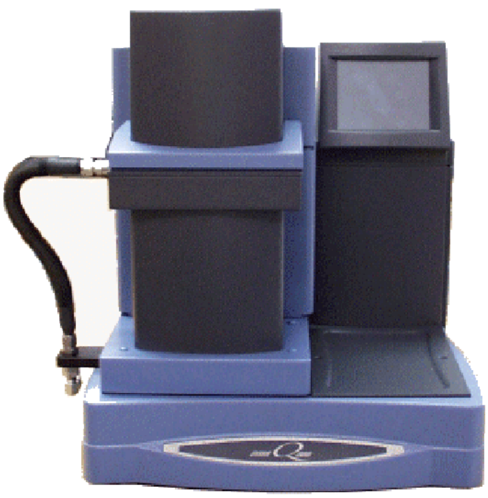

Figure 2.2 Dynamic Mechanical Analyzer.

To acquire and manage the results, this machine uses a software which allows the user to visualize the obtained results, etc. To control the DMA, the user is able to choose between this software and the DMA's touch-screen when configuring test parameters.

Basic instrument characteristics are presented in **Table 2.4** and those focused specifically on our tests in **Table 2.5**.

Table 2.4 Basic instrument characteristics.

Operating environment conditions	Temperature: 15-30 ºC Relative Humidity: 5-80 % (non condensing)
Temperature Range	-145 to 600 ºC
Displacement Range	25 mm
Loading	0,001 to 18 N

Table 2.5 Specific characteristics for AA rectangular samples tests.

Clamp	3 Point Bending
Sample Length	50 mm (distance between support struts)
Sample Width	15 mm (varies slightly for each sample)
Sample Thickness	2 mm (varies slightly for each sample)

2.2.2 Stress-Strain Test

Imagine we take a chewing gum and we begin to stress it at a constant force rate. As the process runs, we are able to see clearly the gum's external structure is suffering changes. Chewing gum could be considered as a rubber.

Although metal compositions as AA respond to Stress-Strain (or ST-ST) tests in a considerably different matter, the previous example can help us to create a first idea of what an ST-ST test is.

The principle is basic: assuming we apply a force that at a certain moment of time is called *F*, the sample will experience a tendency to become stretched. This will create internal reactions known as stresses. We can already introduce the equation seen in expression **(2.1)**.

$$\sigma = \frac{F}{A} \tag{2.1}$$

If we add the variable ε, or strain:

$$d\varepsilon = \frac{dl}{l} \tag{2.2}$$

or, upon integration:

$$\varepsilon = \int_{l_0}^{l_1} \frac{dl}{l} = \ln \frac{l_1}{l_0} \tag{2.3}$$

According to Hooke's Law, if we finally define Young's modulus *E* as:

$$E = \frac{\sigma}{\varepsilon} \tag{2.4}$$

For a 3 Point Bending clamp (which is the one used to perform all the experiments reported in this document), the Stress-Strain model equations for a a sample having a rectangular cross-sectional shape are:

$$\sigma_x = \frac{PLt}{4I} = \frac{3PL}{wt^2} \tag{2.5}$$

and:

$$\varepsilon_x = \frac{6\delta t}{2L^2\left[1 + \frac{6}{10}(1+\nu)\left(\frac{t}{L}\right)^2\right]} \tag{2.6}$$

being:

$$I = \frac{wt^3}{12} \tag{2.7}$$

Aluminium alloys, as they are metals, commonly present a Hookean solid behaviour. As regards to an Stress-Strain test, this means that the ST-ST representation will have a linear response, as shown in **Figure 2.3**.

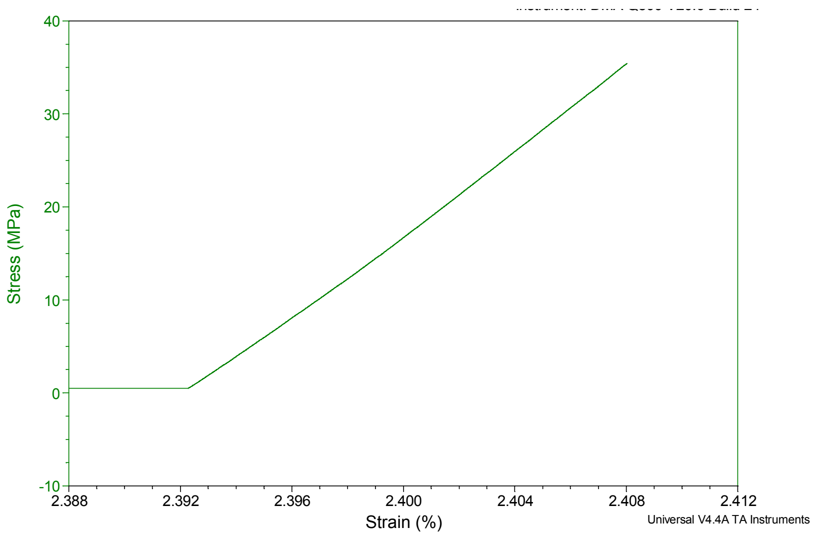

Figure 2.3 Stress-Strain plot for a AA7075 T6 with heat treatment.

2.2.3 Frequency Sweep-Temperature Step Test

Firstly, it is worth to say that FS-TS test is a dynamic analysis, which means that the applied loads are oscillatory (and test may have one or multiple frequencies) and they are applied while the test progresses. On the other, ST-ST test only describes mechanical properties applied at a selected temperature while applying a unique increase in value load.

The Frequency Sweep-Temperature Step Test (FS-TS) consists on a dynamic mechanical test which applies oscillatory forces to the sample, with magnitude and frequencies selected by the user, and measures strain to calculate the storage modulus and loss modulus (see chapter 1).

The start temperature of the test has been always 35ºC. Once reached, the DMA maintains this temperature for 30s while applying the oscillatory forces (these are present during all the process). After this time, the furnace increases its temperature 5ºC more and repeats the process and so up to 375ºC. In this respect it is worth to point out that other values of final temperature were tested: the first series of processes (look at section referring to methodology, below in this chapter) were attempted with a final value of 335ºC. However, in order to increase the amount of information per test this value was increased up to 500ºC. So bad results where obtained that we finally had to consider 375ºC as the maximum temperature for the test.

2.3 Hardness Analysis

During the study of the AA properties hardness values of our samples were required to be known. Lets attempt the key concepts related to this property.

2.3.1 Description

As our samples have macroscopic size, a macroindentation test was selected. To perform macroindentation hardness analysis an *Struers Duramin 1* microdurometer was used (see **Figure 2.4**). The method this instrument uses to compute the samples is by indentation; this means that it computes values of hardness studying the size of the mark resulting by the indentation.

Figure 2.4 Microdurometer.

The results obtained are computed by a software application through which all test parameters can be set or modified, if appropriate.

2.3.2 Vickers (or Diamond Pyramid) Hardness Macroindentation Test

To perform this test, a pyramidal indenter with a square base made of diamond with an angle between faces of 136º is needed. Compared with other indentation tests as the Brinell test, Vickers just needs a unique indenter to cover all materials no matter the value of softness.

A multiple indentation process will give us more accurate results. To proceed the method without interaction between work-hardening regions, it must be taken into account that the distance between indentations must be more than 2.5 times the length between indentation diagonal.

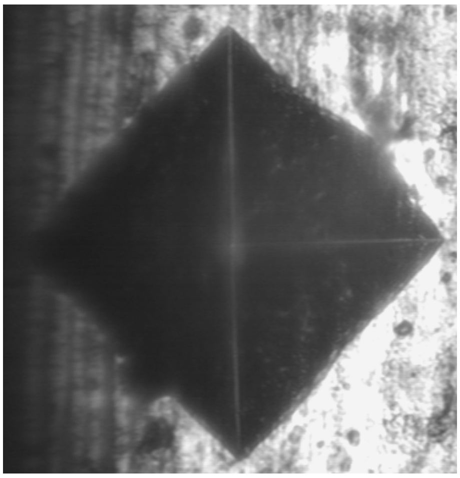

Figure 2.5 HV indentation in AA 7075 T6.

2.4 Differential Scanning Calorimetric Analysis

DSC curves describe heat transmission processes appearing in a process containing a temperature change. For this reason information is presented graphically as a function of heat flux versus temperature.

DSC has just been used to compare them with FS-TS plots and analyze its phase transitions. Any DSC test has been done for the present study.

For this analysis, the endothermic regions are represented by a negative slope rate and exothermic for ascendant lines. See **Figure 2.6**.

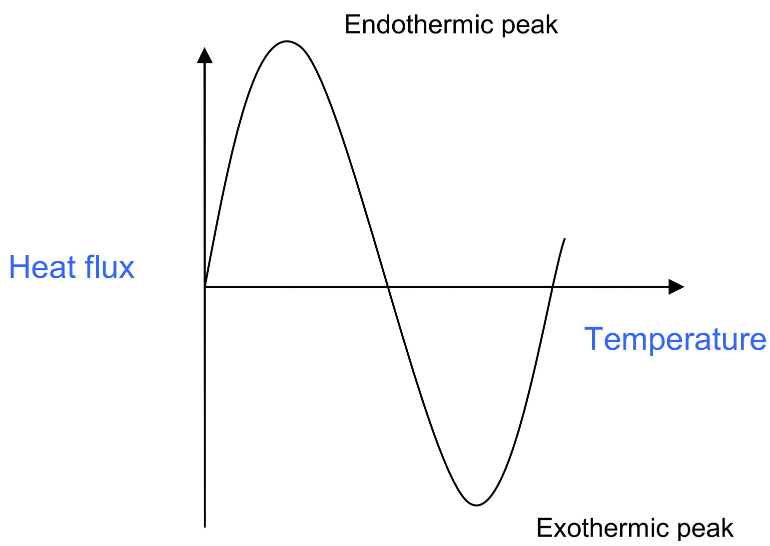

Figure 2.6 DSC endothermic and exothermic regions.

2.5 Methodology

Both AA 2024 T3 and AA 7075 T6 were received in sheet form of 2-mm thickness. Seven samples of each alloy were cut and then submitted to the heat treatments specified before in this document. Heat treatments have been performed with a muffle furnace *Hobersal HD 230* for both retrogression and re-ageing processes.

Figure 2.7 Muffle furnace used in the experimental process.

Once the samples are ready, the test and analysis proceed. To do so, two different instruments have been used: a Dynamic Mechanical Analyzer (DMA) *TA Instruments DMA Q800* will permit us to characterize the viscoelastic properties of each different treatment and a Microdurometer *Struers Duramin-5* has been used to determine the hardness after each process.

Figure 2.8 Introducing a sample into DMA's clamp.

The sequence followed to do the tests has been the same for each sample:

- Perform a FS-TS test to describe its viscoelastic properties.
- Perform a ST-ST test after the FS-TS to compare the change in mechanical properties before and after FS-TS.

It is important to say that in some samples a ST-ST test was also performed before being tested with the FS-TS method. The reason is that the latter test achieves high values of temperatures, which affects considerably the samples as explained in the results analysis made in chapter 3. After doing a complete series of treated samples analysis with three tests performed for either kind of treatment we arrived to the conclusion that only the FS-TS and second ST-ST test contributed to the study. This will be in-depth explained in the following chapter, dedicated to results and discussion.

A total number of two series of treatments has been analyzed: the first one's objective was to become familiar with all the procedure involving the study of the samples and the other's results have been in practical effects considered as the valid ones.

About hardness tests, just have been done to confirm the kind of AA supplied.

Worth to mention is that the levels of precision obtained proceeding heat treatments are quite limited due to:

- The temperature of the oven experiences a deep overshoot, proportional to the rate of increasing you select.
- All of them have been performed putting and extracting the samples by hand in the oven. Applying this method, you have to consider some aspects as the temperature decrement once you open the door for a

relatively short period -which follows an approximate rate of -1ºC/s- as well as the subsequent overshoot the oven temperature will experience due to this procedure.

As well as after samples cutting, a study of errors should be performed in order to reduce the effects resulting on the points detailed above. An interesting conclusion we could arrive to after proceeding this method was that the most susceptible parameter to add errors to the results was L with an order of 2,5.

3 CHAPTER 3. RESULTS AND DISCUSSION

This chapter is dedicated to present the experimental results obtained by the author, as well as discussing them. Special emphasis will be placed in connecting the experimental results with the theoretical explanations found in literature.

As explained in Chapter 2, several series of treatments have been analyzed. The results presented here-in are those obtained for three series of samples: a series of samples made with AA 2024 T3, and 2 identical series of samples from the second series. This is due to the fact that tests on this one were more accurately performed and results are more reliable.

Considerations:

- As said before, ST-ST1 is referred to Stress-Strain tests performed before FS-TS and ST-ST2 is termed for the ones performed after FS-TS.
- To simplify the analysis of some of the results, re-ageing times and temperatures are not taken into consideration: it is supposed that their effect is not comparable to the one due to the retrogression treatments, as reported in literature. The difference between RA temperatures does not exceed 10ºC and maximum difference from the lower to the highest time is 12 hours. We assume these differences do not affect considerably to the results.
- ST-ST1 tests were only performed in AA 7075 T6 samples.

3.1.1 ST-ST tests

ST-ST results will be presented using tables and graphics. In some cases, strain values seem not to be very reliable and/or the plots start at high values of strain. This may be a consequence of experimental errors (for instance, an incorrect positioning of the sample). The method the author uses to analyze the results for this type of tests is by plotting all corresponding *E* values for each treatment in the same graphic, in order to identify the tendencies. This is what follows in this section. In **Figure 3.1** you can see an example of ST-ST plot where *E* would be computed as the slope of the curve.

Note the measured *E* value for AA 2024 T3 without treatment (as-received) is 96,007 GPa, and 153,169 GPa for the AA 7075 T6.

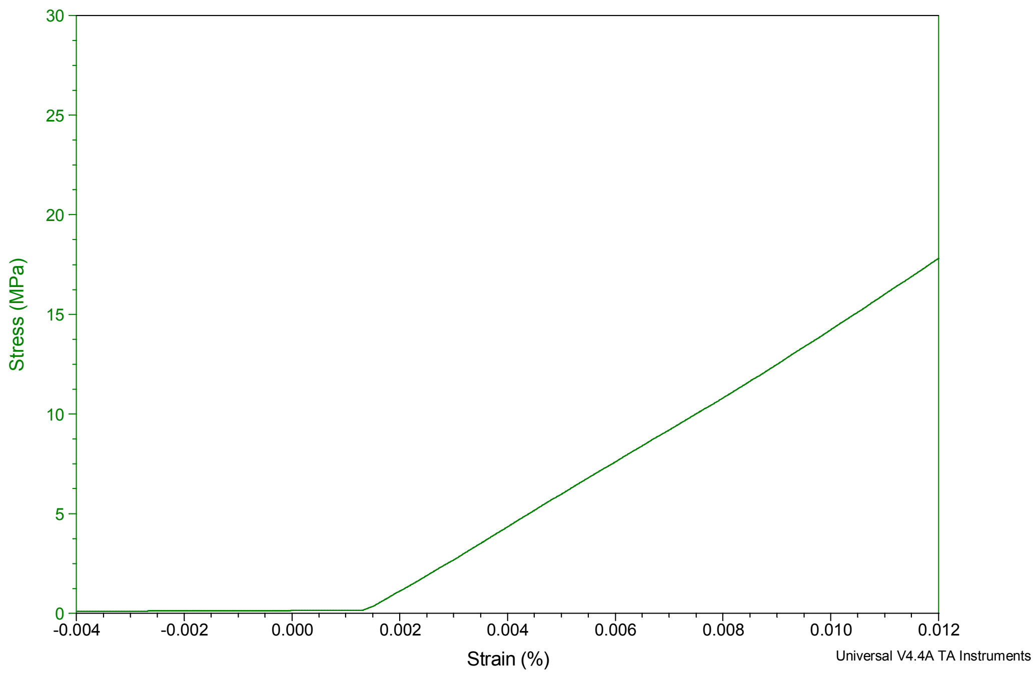

Figure 3.1 AA 2024 T3 ST-ST plot.

3.1.1.1 ST-ST1

These ST-ST1 tests (Stress-Strain tests prior to FS-TS) were only performed on the samples made of AA 7075. Comparing the value of *E* of the untreated sample (153,169 GPa for 7075 T6) with the values obtained for the heat-treated samples, it is observed that all of the heat-treated samples show a decreased value of E.

The results obtained for the various AA 7075 samples through ST-ST1 are plotted in **Figure 3.2**. By simple inspection of this point results in the plot, it seems difficult to find a correspondence between R temperature or time and *E*. To find tendencies, a parametric study should be made in which first only temperature was changed for the various treatments, maintaining the time as a constant. Then, a new series of treatment should be tested in which only time was changed, maintaining the temperature constant.

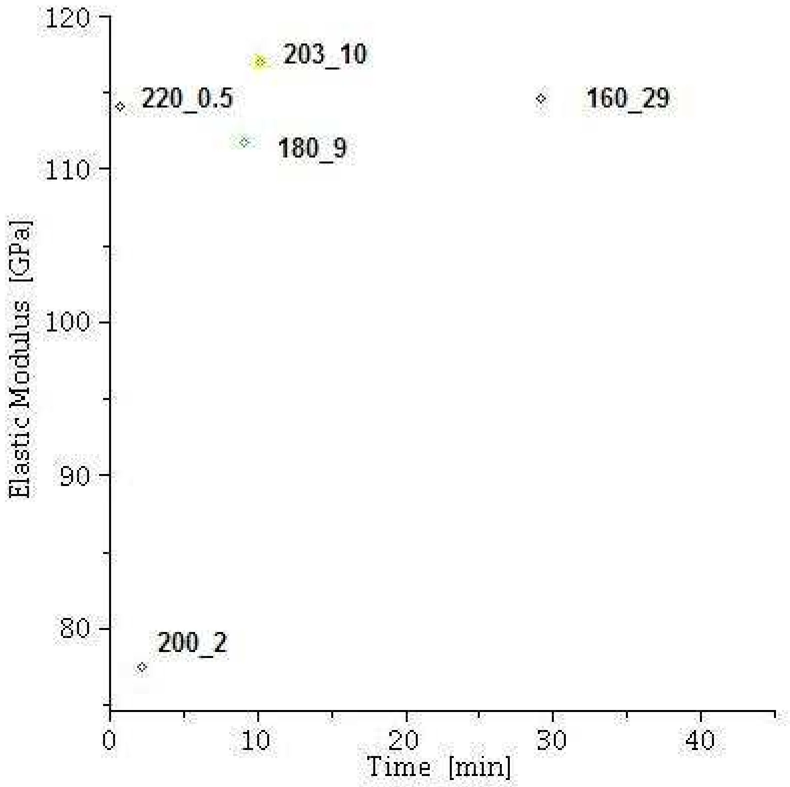

Figure 3.2 ST-ST1 results for heat-treated AA 7075 samples.

3.1.1.2 ST-ST2

Table 3.1 shows the values obtained through ST-ST2 for the samples of each aluminium alloy.

Table 3.1 *E* results for the ST-ST2 test.

Treatment	*E* [GPa]	
	AA 2024	AA 7075
R at 160ºC for 29 min and RA at 120ºC for 38h	195,413	85,853
R at 180ºC for 9 min and RA at 120ºC for 38h	59,217	122,131
R at 180ºC for 40 min and RA at 130ºC for 12h	105,382	71,272
R at 200ºC for 2 min and RA at 120ºC for 24h	96,189	142,816
R at 200ºC for 5 min and RA at 120ºC for 24h	106,086	148,112
R at 200ºC for 30 min and RA at 130ºC for 12h	121,680	101,635
R at 220ºC for 0,5 min and RA at 120ºC for 24h	113,137	177,985

These results are plotted in **Figure 3.3** and **Figure 3.4**.

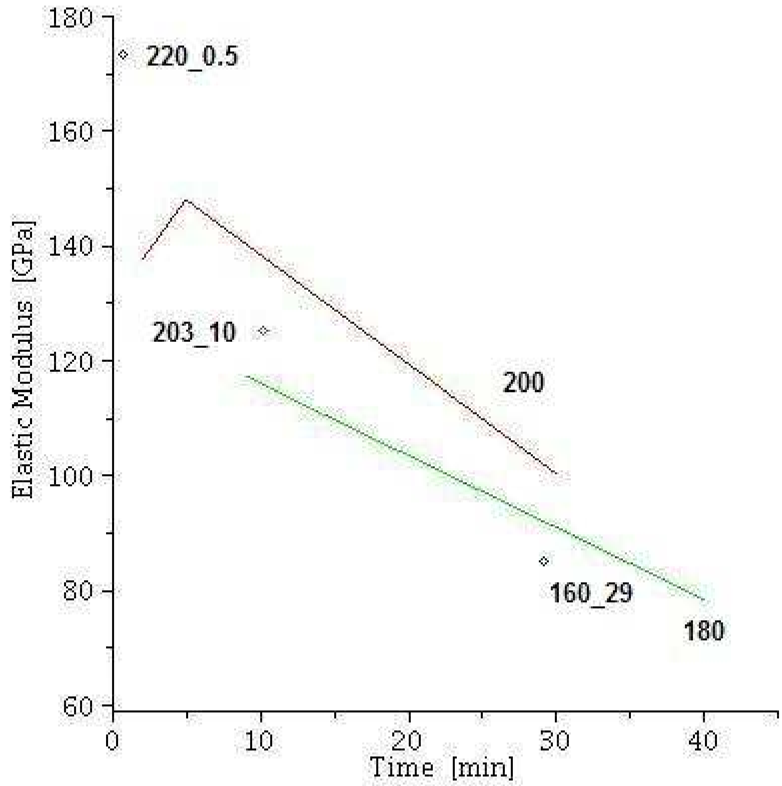

Figure 3.3 ST-ST2 results for heat-treated AA 7075 samples.

Comments of ST-ST2 results for heat-treated AA 7075 samples

By comparison of the values obtained by ST-ST1 and ST-ST2 tests for AA 7075 (**Figure 3.2** and **Figure 3.3**), a general increase in *E* value is observed for the latter. Exception is made for the sample in which the R temperature and time were 160ºC and 29 min, respectively.

In this plot and in the following, polygonal lines join points corresponding to the representation of R treatments having different times but having the same temperature values. Namely, in the plot above, the green line joins the points of *E* for the R treatments at 180ºC and 9 and 40 min, and the red line joins the points of *E* for the R treatments at 200ºC and 2, 5 and 30 min.

If attention is focused in these polygonal lines, we can see a decrease in *E* as long as the R time increases. Exception is made for the sample in which the R temperature and time were 200ºC and 5 min, respectively.

Concerning the R temperature in this plot, it seems that an increase of the temperature of the treatment leads to an increase in the Elastic modulus. Again, the sample retrogressed at 160ºC for 29 minutes, and also the one retrogressed at 203ºC for 10 minutes, may seem not to follow this tendency completely, although much more tests and points are needed to ascertain all these observations.

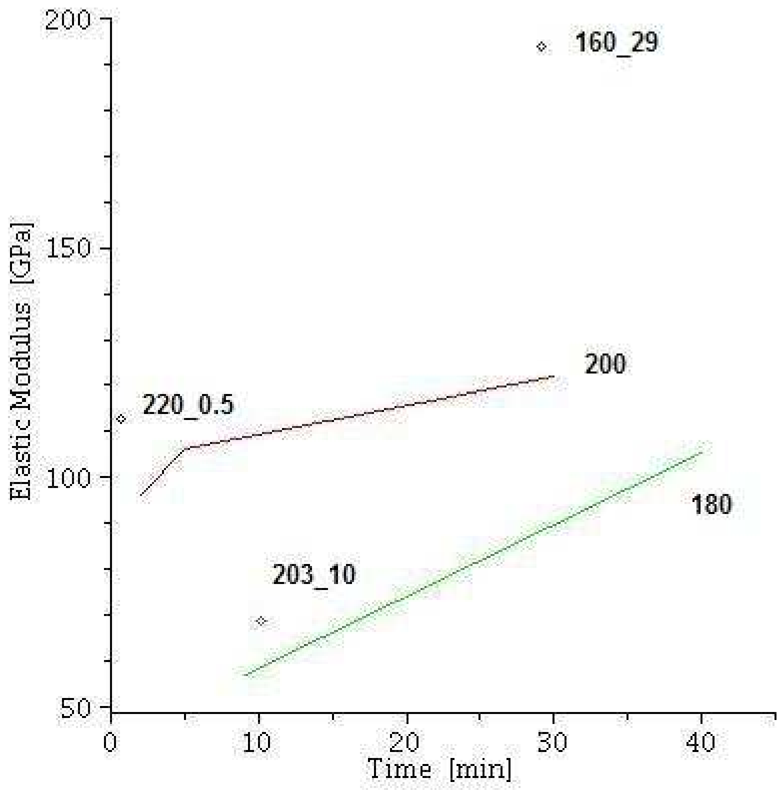

Figure 3.4 ST-ST2 results for heat-treated AA 2024 samples.

Comments of ST-ST2 results for heat-treated AA 2024 samples

In this case, the experimental results show on one hand that, as we increase the R time, also the value of *E* increases (see red and green line). On the other hand, we see that *E* increases when having higher values of temperature R. Once again, the samples retrogressed at 160ºC for 29 minutes and at 203ºC for 10 minutes seem not to follow any of these reported tendencies.

3.1.1.3 Discussion

E varies for each different treatment. As pointed out above, *E* for AA 2024 T3 heat-treated samples acquire higher values than for non-treated ones but it is not the case for AA 7075 T6 ones. This could be explained theoretically because the R treatments modify considerably the microstructure of the material as explained in chapter 1.

3.1.2 FS-TS tests

Respect to FS-TS results, some of the signals that characterize the viscoelastic behaviour are the Storage and Loss moduli. The first provides information about the stored deformation energy. This energy is released upon unloading This modulus is linked to an elastic behaviour. The Loss modulus allows us to estimate the amount of energy that is lost as frictional heat, which is closely related to a viscous response.

After analyzing the results, it can be observed that the storage modulus values are generally higher initially and gradually diminish as the temperature of the test increases. The loss modulus for all samples generally increases with temperature, from a starting point at lower values than the storage modulus.

To analyze FS-TS plots it is interesting to compare them with DSC curves. This kind of analysis, as explained before, represents the changes in energy by analysing the heat rate in the process. We will use this method for AA 7075 T6 results analysis.

As seen in **Figure 3.5**, T6 treatment presents a first endothermic peak between 150ºC and 230ºC followed by a several exothermic peaks finishing with a wide endothermic zone up to 460ºC.

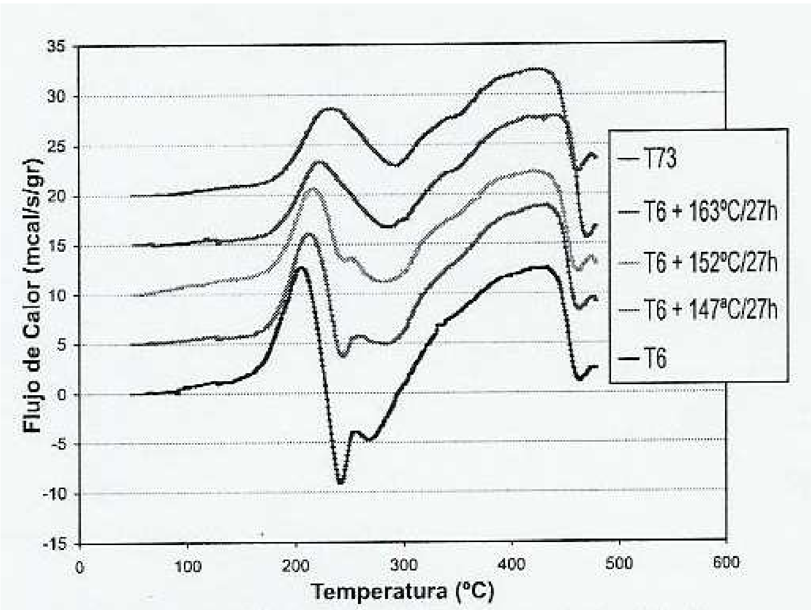

Figure 3.5 DSC curves for AA 7075 with different treatments. [10]

Comparing these previous DSC results with the results obtained by FS-TS test, there is a clear change in the Storage modulus and Loss modulus slopes at 150ºC which probably corresponds to the phase transformation related to the endothermic peak seen in **Figure 3.5**. At approximately 250ºC, the slope in **Figure 3.6** changes again, which may correspond to the exothermic period in the DSC diagrams reaching values of about 300ºC. From now on, all the FS-TS plots show a characteristic change in the slopes of the Storage modulus and Loss modulus, once again corresponding to the endothermic region in the DSC diagram.

Note that for both *E'* and *E''* three lines are shown. Each one of them is for a different frequency value (1, 10 and 12 Hz from lower to upper).

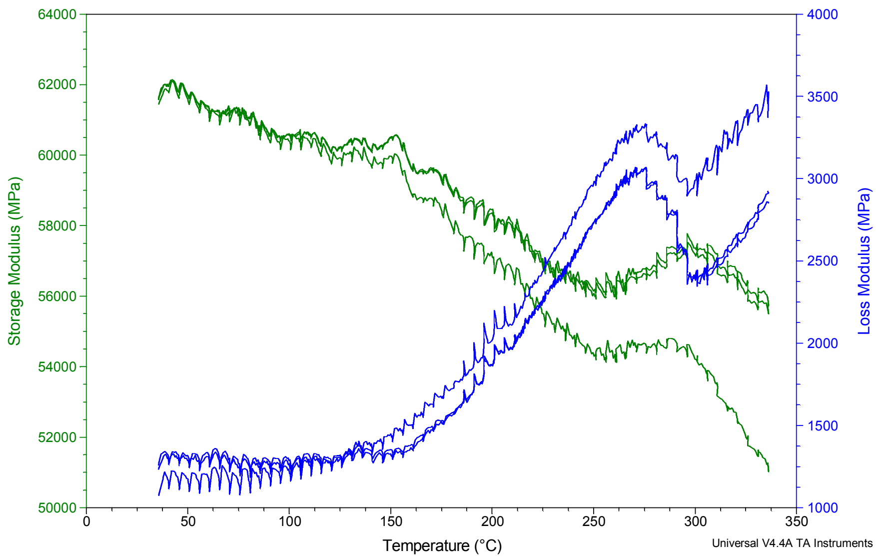

Figure 3.6 FS-TS AA 7075 T6.

3.1.2.1 AA 2024 T3 plot analysis

This section presents results plotted comparing two similar heat treatments. Heat treatments are shown with the format T_t (e.g. 160_29 reffers to a R temperature of 160ºC for 29 min).

By simple inspection of the plots, we will focus on the following parameters to compare each pair:

- Temperature when plots of different frequency get separated.
- The changes in slope and its corresponding temperature.

Note that *E'* and *E''* values are not considered. The results analysis has a descriptive motivation.

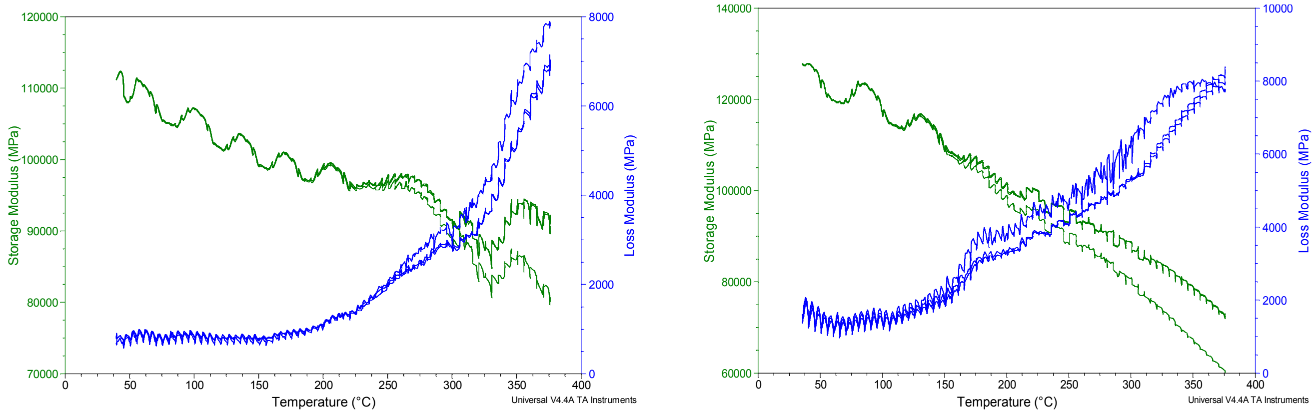

Figure 3.7 Plots for 2024 160_29 and 2024 180_40.

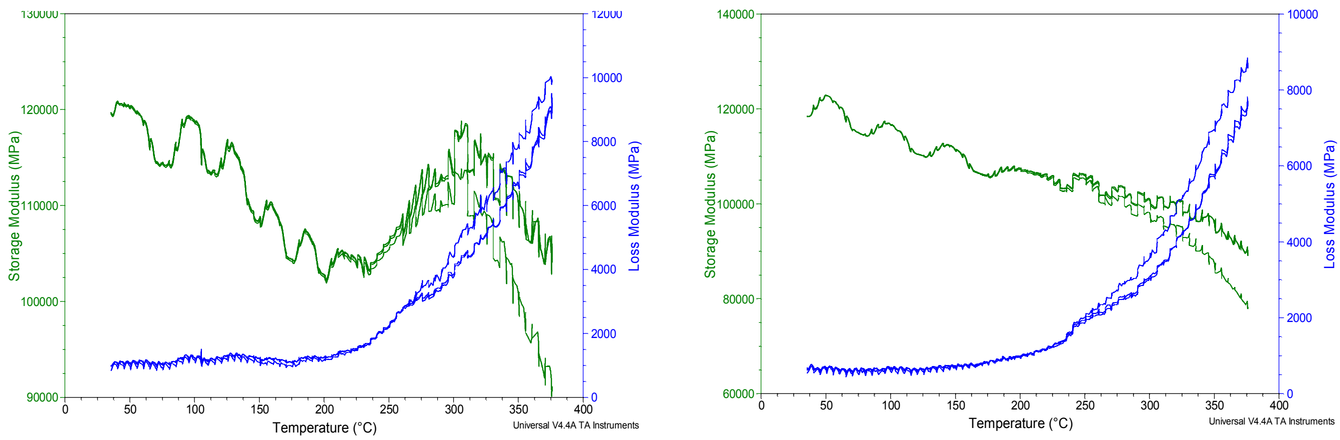

Figure 3.8 Plots for 2024 200_5 and 2024 200_30.

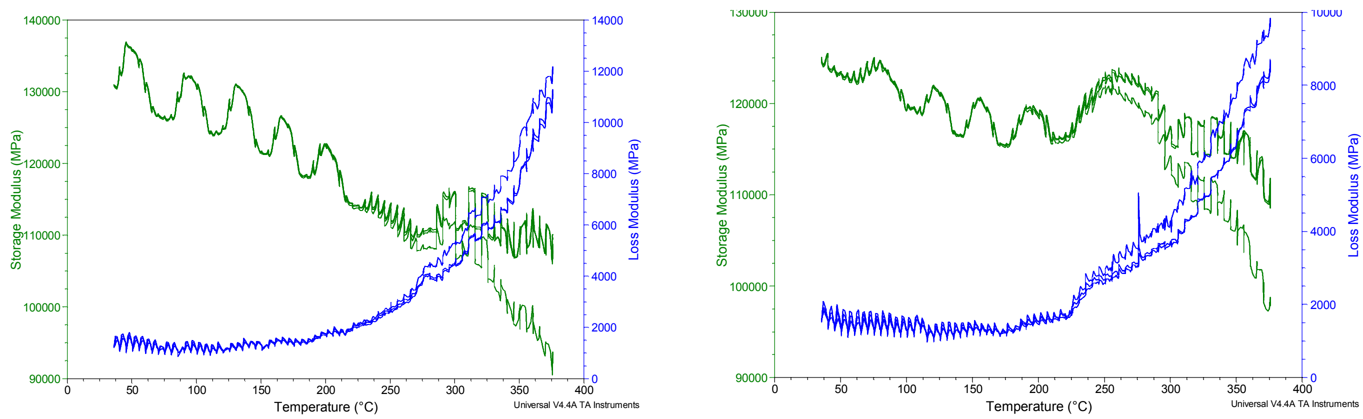

Figure 3.9 Plots for 2024 203_10 and 2024 220_0.5.

The division of different frequency lines appears between 200ºC and 250ºC for all the heat treatments except for 203_10 and 180_40. If we analyze the changes in Storage modulus’ slope, 180_40 and 200_30 seems not to change abruptly for high frequencies while in the rest of treatments a change in slope is shown both for low and high frequencies (beginning with a negative slope, changing to positive one and again decreasing in *E’*).

3.1.2.2 AA 7075 T6 plot analysis

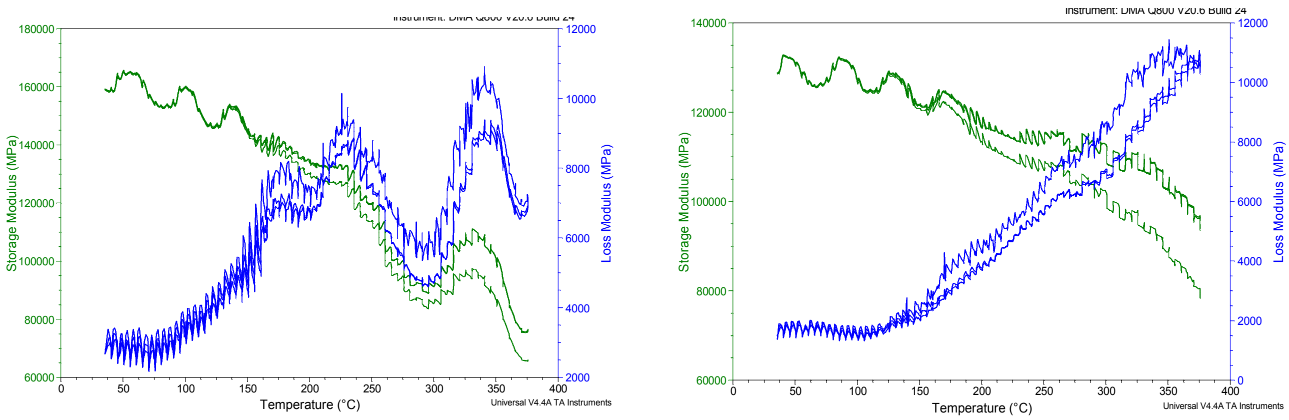

Figure 3.10 Plots for 7075 160_29 and 7075 180_40.

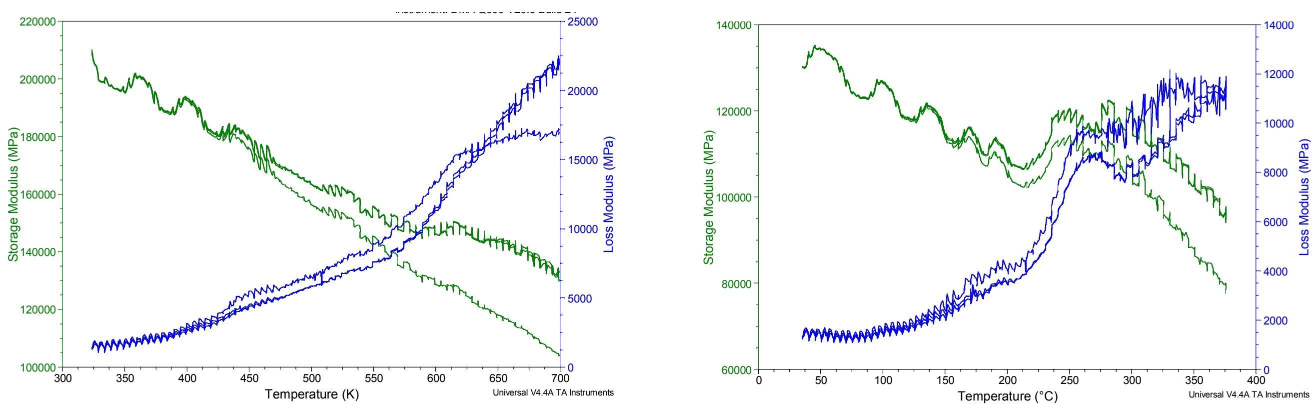

Figure 3.11 Plots for 7075 200_2 and 7075 200_5.

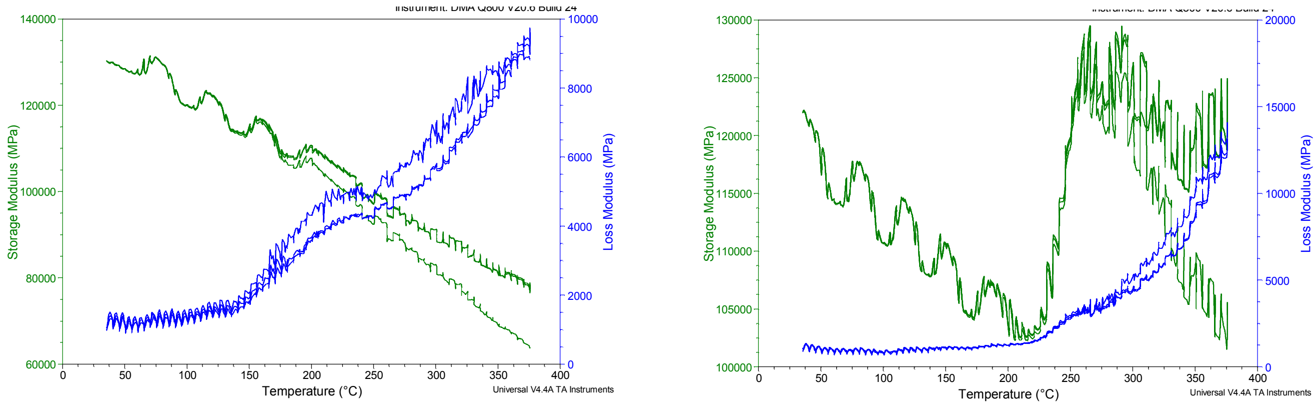

Figure 3.12 Plots for 7075 200_30 and 7075 203_10.

For the case of AA 7075, the division of frequency lines generally occurs before than in 2024 (around 150ºC except for 203_10). For 200_2 and 200_30's *E'* seems to decrease uniformly for both low and high frequencies while the other plots show different slope changes also for all frequencies range.

3.1.2.3 Discussion

The variations in mechanical properties are the result of changes in the precipitates and the microstructure of the samples. Also the viscoelastic properties seem to correlate well to changes in the microstructure.

As reported in previous studies, for AA 7075 T6 the first endothermic peak at low temperature (around 200ºC) corresponds to the dissolution of *GP zones* as well as *η* present in matrix microstructure. Exothermic regions for solution treated elements have usually two peak zones: the first one is due to the formation of *η'* from pre-existing *GP zones* and the second corresponds also to a formation but in this case of *η* from *η'*.

If we compare 2024 and 7075 FS-TS results for the same treatments, we appreciate that for the cases of 180_40 and 200_30 the *E'* lines seem to describe a similar profile while it is not the same for 200_5 and 203_10 cases.

Once analyzing the results it seems that the higher the frequency of the forces applied in tests, the more elastic behaviour. Note that in the whole series of test slight and continuous peaks are obtained for low temperatures which could be caused by the application of the load during the FS-TS test.

3.1.3 Vickers (or Diamond Pyramid) Hardness Macroindentation Test

As said before, the ageing process usually strengthens the material we deal with and can be also made at room temperature. Due to this, the first samples we analyzed were probably naturally aged before testing them.

For this reason, HV values higher than expected were registered. In **Table 3.2** comparison between hardness values for as-received plates and after three years at room temperature are presented.

Table 3.2 Hardness values for as-received and aged plates.

AA	Hardness as-received [HV]	Hardness after three years (F=19,6N) [HV]
7075 T6	129.5	160,1
		154,2
		156,2
		157,5
		156,7
2024 T3	169.5	198,3
		197,9
		198,5
		196,9
		197,9

4 CHAPTER 4. CONCLUSIONS

In this study we have considered three different types of tests related to the study of aluminium's properties. The tests were:

- Stress-strain test.
- Frequency sweep-temperature step test.
- Vickers hardness macroindentation test.

These tests were performed on three series of samples. Furthermore, the whole preparation of these samples from the as-received aluminium plates, including the machining as well as the heat treatments, was performed by the author.

General conclusions obtained:

- Microstructural phases during a heat process are, for AA 7075 T6 (for AA 2024 T3 θ' instead of *η'* and θ instead of *η*):

  SSS → GP zones → η' → η → matrix in equilibrium

- The higher the R temperature, the more stable the Al matrix after RA.
- RA stabilizes Al's microstructure.
- At some temperature value, high and low frequencies' plot for *E'* and *E''* divides.
- Some oscillations appear generally in *E'* plots at low temperature values.
- Some uniform oscillations appear generally in *E''* at low temperatures shorter in modulus than for the case of *E'* and possibly due to the load apply in the process.
- The higher the frequency of the forces applied in tests, the more elastic behaviour.

Obtained values for hardness were coherent, as explained in its corresponding results section, but it is not the case for *E*, *E'* and *E''* which were generally expected to be lower. A correspondence between these obtained results is appreciated so the cause of that may be attributed to an instrument error.

As the author knows, few number of studies referred to Al's viscoleasticity have been done. This fact has both pros and cons. On the one hand, this usually reveals a wide range of curious and undiscovered results or conclusions. On the other hand, it makes more difficult to ascertain if your work has followed the right path or even followed any.

Nevertheless, a primary attempt in study of viscoelastic properties in metals and concretely aluminium’s has been performed. Due to the modesty of this document it can not be affirmed that we have opened a door in the field but it may be considered as a step forward in that direction.

An exhaustive control of the hours invested in this study has been done. **Table 4.1** presents a feasible income for an engineer in practice with a corresponding salary of 8€/worked hour.

Table 4.1 Basic estimation of income.

Activity	Income [€/worked hour]	Total income [€]
420 worked hours	8	3.360

As a future work, it would be interesting to study and test samples for wider ranges of frequency of the forces applied. Once analyzing the results, this has been an interesting point to take into account.

Furthermore, the results presented in this document may be in-depth analyzed to extract further information about viscoelatic properties referring both studied AA or even as a support for related studies.

5 ACRONYMS AND SYMBOLS

A: ageing
3PB: 3-Point Bending
AA: aluminium alloy
Al: aluminium
DMA: Dynamic Mechanical Analyzer
DSC: Differential Scanning Calorimetry
E: elastic modulus
FS-TS: frequency sweep-temperature step
HV: Vickers Hardness
L: length
R: retrogression
RA: re-ageing
RRA: retrogression and re-ageing
Quenching: brusque cooling of the samples after a heat treatment process, usually at room temperature
SCC: stress corrosion cracking
Solution: homogeneous mixture of two or more substances
SSS: supersaturated solid solution
ST-ST: stress-strain

E’ : storage modulus [Pa]
E” : loss modulus [Pa]
HV: vickers hardness [dimensionless]
F: applied load [N]
d: measured length of the diagonal of the indentation [mm]
w: sample width [mm]
σ : stress[Pa]
A: cross-sectional area[m2]
ε : strain[%]
l : longitude[m]
P: applied force[Pa]
δ : amplitude of deformation[mm]
L: 1/2 sample length (span)[mm]
t: sample thickness[mm]
w: sample width[mm]
ν : poison’s ratio[dimensionless]
I: moment of inertia (for a rectangular shaped sample)[Nm]

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