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Onset frequency of fatigue effects in pure aluminum and 7075 (AlZnMg) and 2024 (AlCuMg) alloys

Jose I. Rojas [1,*] and Daniel Crespo [2]

Received: date; Accepted: date; Published: date
Academic Editor: name

[1] Department of Physics - Section of Aerospace Engineering of Escola d'Enginyeria de Telecomunicació i Aeroespacial de Castelldefels, Universitat Politècnica de Catalunya; c/ Esteve Terradas 7, 08860, Castelldefels (Spain); Correspondence: josep.ignasi.rojas@upc.edu
[2] Department of Physics, Universitat Politècnica de Catalunya; c/ Esteve Terradas 7, 08860, Castelldefels (Spain); daniel.crespo@upc.edu
* Correspondence: josep.ignasi.rojas@upc.edu; Tel.: +34-93-413-4130

Abstract: The viscoelastic response of pure Al and 7075 (AlZnMg) and 2024 (AlCuMg) alloys, obtained with a dynamic-mechanical analyzer (DMA), is studied. The purpose is to identify relationships between viscoelasticity and fatigue response of these materials, of great interest for structural applications, in view of their mutual dependence on intrinsic microstructural effects associated to internal friction. The objective is to investigate the influence of dynamic loading frequency and temperature on fatigue, based on their effect on the viscoelastic behavior. This research suggests that the decrease of yield and fatigue behavior reported for Al alloys as temperature increases may be associated to the increase of internal friction. Also, materials subjected to dynamic loading below a given threshold frequency exhibit a static-like response, such that creep mechanisms dominate and fatigue effects are negligible. In this work, an alternative procedure to the time-consuming fatigue tests is proposed to estimate this threshold frequency, based on the frequency dependence of the initial decrease of storage modulus with temperature, obtained from the relatively short DMA tests. This allows for a fast estimation of the threshold frequency. The frequencies obtained for pure Al and 2024 and 7075 alloys are 0.001–0.005, 0.006 and 0.075–0.350 Hz, respectively.

Keywords: aluminum alloys; AlZnMg; AlCuMg; viscoelasticity; dynamic-mechanical analysis; internal friction; loading frequency; fatigue

1. Introduction

Fatigue is a form of failure which may occur in structures subjected to dynamic loading, even at stress levels significantly lower than the ultimate tensile strength under static loading [1]. Failure results from a gradual process of damage accumulation and local strength reduction, which is manifested by crack initiation and propagation, after relatively long periods of dynamic loading. It is particularly dangerous in structural applications because of its brittle, catastrophic nature, and because it occurs suddenly and without warning, since very little plastic deformation is observed in the material prior to failure [1,2]. The fatigue fracture behavior of materials is dominated by the microstructure [3]. When a material is subjected to dynamic loading, energy is dissipated due to internal friction phenomena. Most of this energy manifests as heat and causes temperature increases in the material, a process termed hysteresis heating. It has been suggested that all metals, when subjected to hysteresis heating, are prone to fatigue [4].

In previous investigations, the viscoelastic response (including the internal friction behavior) of Al alloys (AA) 7075 and 2024 was measured with a dynamic-mechanical analyzer (DMA) [5,6]. In this work, experimental results on the viscoelastic response of pure Al are presented, first. Second, these results for pure Al and the aforementioned alloys are analyzed with the purpose of identifying relationships between the viscoelastic response and the fatigue behavior of these materials, in view of their mutual dependence on intrinsic microstructural effects associated to internal friction [7]. Particularly, the objective is to investigate the influence of the dynamic loading frequency and temperature on fatigue, based on the effect of these variables on the viscoelastic behavior. The results seem to support the work by Amiri and Khonsari [4], as per the correlation between the fatigue life and the initial hysteresis heating during dynamic loading. Namely, it is likely that the decrease of yield and fatigue response observed in some metals as temperature increases is associated to the increase of internal friction with temperature. Moreover, following previous investigations by other researchers, suggesting the existence of a threshold frequency marking the transition from a static-like response of the material to the advent of fatigue effects, in this work an alternative procedure is proposed to estimate this threshold frequency based on experimental data obtained with the relatively short DMA tests. These findings are of remarkable importance, especially for the alloys, in view of their widespread use in structural applications under dynamic loading. Particularly, AA 7075 and 2024 are key representatives of the AlZnMg and AlCuMg alloy families (or 7xxx and 2xxx series, respectively), belonging to the group of age-hardenable alloys. These alloys feature excellent mechanical properties and are highly suitable to a number of industrial applications, especially in the aerospace sector and transport industry [8].

1.1. Influence of the loading frequency on the fatigue response of metals

Fatigue may be sensitive, for instance, to the strength, the manufacturing conditions and the surface treatment of the material, but also to the loading frequency and loading environment, the displacement rates and the stress amplitude [1,9–11]. In this work, we address the effects of the dynamic loading frequency and temperature, in conjunction with the microstructure.

Much research has been devoted to ascertain whether accelerated laboratory tests (i.e., with loading frequencies higher than those in service conditions) affect the fatigue response and how, but this is yet a controversial issue. This is particularly true for the study of high cycle fatigue (HCF) and very high cycle fatigue (VHCF) behavior by means of very high frequency tests. Tests in VHCF and very low crack growth rates are time consuming with conventional fatigue testing techniques, like rotating bending, with a maximum frequency of 100 Hz. Hence, accelerated laboratory tests are very interesting because a significant reduction of testing time is possible using high-speed servo-hydraulic machines [12], which may work at frequencies of 600 Hz, or specially using ultrasonic equipment, which may reach frequencies of 20 kHz [13].

Zhu et al. [14] state that environmental effects need to be considered, and Mayer et al. [15] explain that this is so because the time-dependent interaction with the environment may cause an extrinsic frequency influence on fatigue properties, on top of the intrinsic strain rate effects. Furuya et al. [12] state that frequency generally affects high frequency fatigue tests because: 1) fatigue limits and lives decrease due to the temperature increase caused by plastic deformation [16]; 2) dislocations may not match the applied frequency because dislocation movement is slow compared to sonic velocity [17]; 3) provided that embrittlement by hydrogen diffusion had an effect [18], fatigue lives would depend on both the number of loading cycles and time. However, Mayer [19] reported also that the HCF behavior of metallic alloys is relatively insensitive to the test frequency, provided that the ultrasonic testing procedure is appropriate (e.g., adequate cooling) and that fatigue-creep interaction and the time-dependent interaction with the environment are negligible. The reasons suggested are, on the one hand, that cyclic plastic straining is limited near the fatigue limit or the threshold of fatigue crack growth (FCG), and thus plastic strain rates are low even at high frequencies; and, on the other hand, the fact that shear stress has little sensitivity to strain rate [20]. Mayer et al. [15] also commented that the influence of frequency becomes significantly smaller if the dynamic stress amplitude is lower, maybe because cyclic loading is almost perfectly elastic.

For body-centered cubic (bcc) metals and metallic alloys, the HCF behavior is reported to be more sensitive to frequency than for face-centered cubic (fcc) metals [13]. However, Furuya et al. [12] observed that fatigue behavior of high-strength steels is independent of frequency. The argued cause was their extremely high-strength, and thus reduced plasticity and dislocation mobility. The hysteresis energy is low in low plasticity materials, and thus the frequency effects on fatigue associated to the temperature increase are minimized. Likewise, Yan et al. [21] observed very little variation of the fatigue strength of high-strength steel when testing at a conventional frequency (52.5 Hz) and at an ultrasonic frequency (20 kHz).

For an Al alloy similar to AA 7075 tested in the HCF regime at room temperature (RT), samples tested at 100 Hz were reported to fail earlier than those tested at 20 kHz. However, the effect of frequency on fatigue behavior was not statistically significant [15]. On the contrary, for E319 cast Al alloy at 293, 423 and 523 K, fatigue life at 20 kHz was 5 to 10 times longer than that at 75 Hz [14], but this author states that fatigue crack initiation is not influenced either by temperature or frequency. Rather, the observed difference in fatigue life is attributed to environmental effects on FCG rate. The fact that the moisture of ambient air deteriorates the fatigue life of high-strength Al alloys by increasing the FCG rate has also been suggested by other authors [15,22]. Namely, Menan and Henaff [23] suggest for AA 2024-T351 that fatigue and corrosion may interact such that FCG rates are enhanced. These synergistic effects are more notorious at low frequencies, for a given number of cycles at RT. Finally, Benson and Hancock [24] observed strain rate effects on cyclic plastic deformation of AA 7075-T6, provided that cyclic stresses were close to the yield stress.

As per low frequency loading, on one hand, Nikbin and Radon [25] proposed a method to predict the frequency region of interaction between creep and FCG using static data (obtained at 423 K for Al alloy RR58) and RT high frequency fatigue data, and assuming a linear cumulative damage law. The results showed that the interaction region is 0.1 to 1 Hz for the Al alloy (see Figure 4 in [25]). In the intermediate (steady state) stage of cracking for static and low frequency tests, crack growth is sensitive to frequency and the fracture mode is time-dependent inter-granular in nature, suggesting that creep dominates. Conversely, for high frequency tests, crack growth is insensitive to frequency and the fracture mode is trans-granular, suggesting that pure fatigue mechanisms dominate. The results indicated also little interaction between these processes.

On the other hand, Henaff et al. [26] analyzed creep crack growth (CCG) rates, FCG rates and creep-fatigue crack growth (CFCG) rates of AA 2650-T6 at 293, 403 and 448 K, for frequencies of 0.05 and 20 Hz. The objective was to enable prediction of crack growth resistance of that alloy under very low frequency loading at elevated temperatures. It was concluded that, in the studied frequency range, frequency has only a slight effect on FCG rates at 448 K. In particular, under low frequency loading it was observed a high increase in fracture surface fraction of inter-granular type, similar to that corresponding to CCG. This shows that creep damage might occur during loading at low frequency, in accordance with findings in [25]. Henaff et al. [26] reported also that, for a given temperature, CFCG is unaffected by frequency above a critical value of the loading frequency (see Figure 12(b) in [26]). Below, CFCG is inversely proportional to excitation frequency, i.e., a time-dependent crack growth processes take place. This researcher suggested the existence of creep-fatigue-environment interaction, as CFCG is affected by the environment at low frequency loading, and proposed an alternative method to predict CFCG rates at very low frequencies, using a superposition model and results obtained at higher frequencies.

1.2. Influence of temperature on the fatigue response of Al alloys

For E319 cast Al alloy tested at 293, 423 and 523 K, Zhu et al. [14] observed that the fatigue strength decreases with temperature, and that the temperature dependence of the fatigue resistance at 108 cycles follows closely the temperature dependence of the yield and tensile strength for this alloy. Furthermore, by integration of a universal version of a modified superposition model, the effects of temperature, frequency and the environment on the S-N curve of this alloy can be predicted, and it is possible also to extrapolate ultrasonic data to conventional fatigue behavior [14]. Henaff et al. [26] concluded that the temperature has almost no influence on FCG rates for AA 2650-T6, after conducting tests at 293, 403 and 448 K and frequencies of 0.05 and 20 Hz. Amiri and Khonsari [4] state that the initial slope of the temperature rise due to hysteresis heating observed at the beginning of fatigue tests is a characteristic of metals. Capitalizing on this, they developed an empirical model that predicts fatigue life based on that slope, thus preserving testing time. Indeed, correlation of the temperature evolution with fatigue has been used successfully in many ways, aside from for predicting fatigue life, as in the previous example. Namely, it has been used for providing information on FCG [27] and the endurance limit of materials [28], or for quantification of the cumulative damage in fatigue [16]. Also, the heat dissipated during ultrasonic cycling can be used to calculate the cyclic plastic strain amplitude [15].

1.3. Influence of the microstructure on the fatigue response of Al alloys

There is abundant research in the literature on the effect of the microstructure on the fatigue response of materials. For example, it is proposed that the mechanisms responsible for the fatigue fracture behavior are associated to competing and synergistic influences of intrinsic microstructural effects and interactions between dislocations and the microstructure [3]. Indeed, researchers claim that prediction of fatigue life should be possible based on the knowledge of the microstructure prior to beginning of service, without the need for expensive, time consuming fatigue experiments [29]. This would enable optimization of the material properties by controlling the microstructure. Accordingly, a model based on dislocation stress was proposed to predict S-N curves using microstructure/material sensitive parameters instead of constitutive equation parameters [29]. The model is successful for low cycle fatigue life prediction.

2. Materials and Methods

The tested specimens were machine cut from sheet of as-received pure Al (99.5 wt.% purity according to the supplier, Alu-Stock, S.A.) in the H24 temper. The H24 temper consists in cold-working (i.e., strain hardening) beyond desired hardness, followed by a softening treatment consisting in annealing up to halfway of peak hardness. The specimens were rectangular plates 60 mm long, 8–12 mm wide and 2 mm thick. Half of these plates were annealed at 750 K for 30 min, and immediately quenched in water to RT, to remove the strain hardening. A TA Instruments Q800 DMA was used to measure the viscoelastic response of the samples in N_2 atmosphere. Namely, the DMA measured the storage modulus E' (i.e., the elastic –real– component of the dynamic tensile modulus, accounting for the deformation energy stored by the material), the loss modulus E'' (i.e., the viscous –imaginary– component of the dynamic tensile modulus, accounting for the energy dissipation due to internal friction during relaxation processes) and the loss tangent (also termed mechanical damping or $\tan\delta$) [7]. The 3-point bending clamp was used, and the DMA was set to sequentially apply dynamic loading with frequencies ranging from 1 to 100 Hz, at temperatures from RT to 723 K in step increments of 5 K. More details on the procedure, as well as the viscoelastic data of AA 7075-T6 and 2024-T3 used in this work, can be found in [5,6].

3. Results and Discussion

3.1. Storage modulus

Figure 1(a) shows E' for pure Al in the H24 temper, from RT to 648 K, while Figure 1(b) shows E' for pure Al, from RT to 723 K, in both cases as obtained from DMA tests at frequencies ranging from 1 to 100 Hz. The behavior of E' for pure Al is similar in some aspects to that observed for AA 7075-T6 and 2024-T3 [5,6] and AA 6082 [30] (an excel file including the values of E', E'', and loss tangent measured for pure Al in the H24 temper, pure Al, AA 7075-T6 and AA 2024-T3 is provided as supplementary material). For example, E' also decreases initially. The slope at low temperatures (below the beginning of the dissolution of Guinier-Preston (GP) and Guinier-Preston-Bagariastkij (GPB) zones, for the alloys) is what is most interesting in this study, as explained in Section 4.3. Also, a significant decrease in E' is observed, with the beginning of this drop shifted to higher temperatures (from around 423 to 523 K) as the loading frequency increases. Thus, at a given temperature, the alloys show a stiffer response (i.e., E' is larger) at higher frequencies, as expected. Also, E' depends more significantly on frequency at high temperatures (above 423 K). The fact that the viscoelastic behavior becomes more prominent with temperature has already been observed in amorphous alloys [31], aside from AA 7075-T6 and 2024-T3 [5,6] and AA 6082 [30].

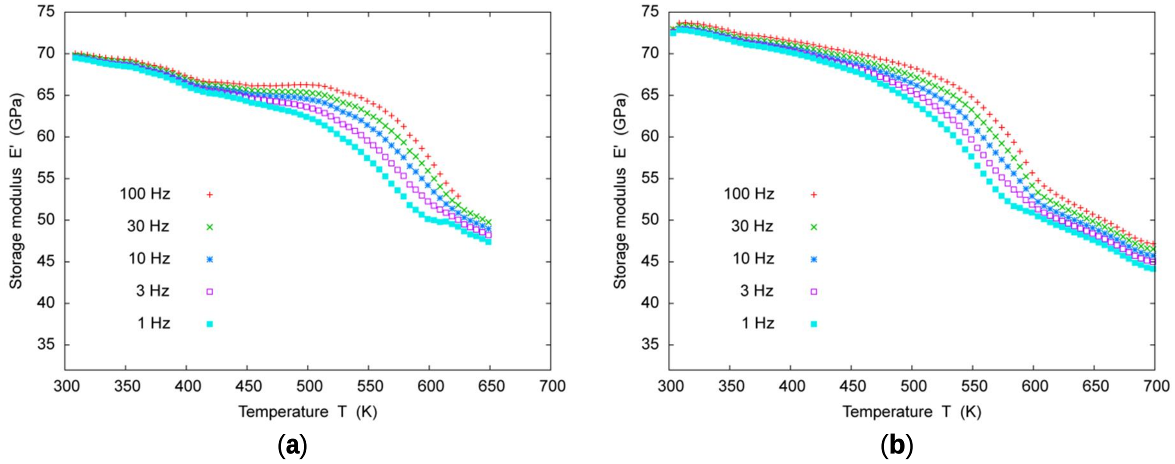


Figure 1. Storage modulus E' vs. temperature T from dynamic-mechanical analyzer tests at frequencies ranging from 1 to 100 Hz: (a) for pure Al in the H24 temper, from room temperature (RT) to 648 K; (b) for pure Al, from RT to 723 K.

3.2. Loss modulus

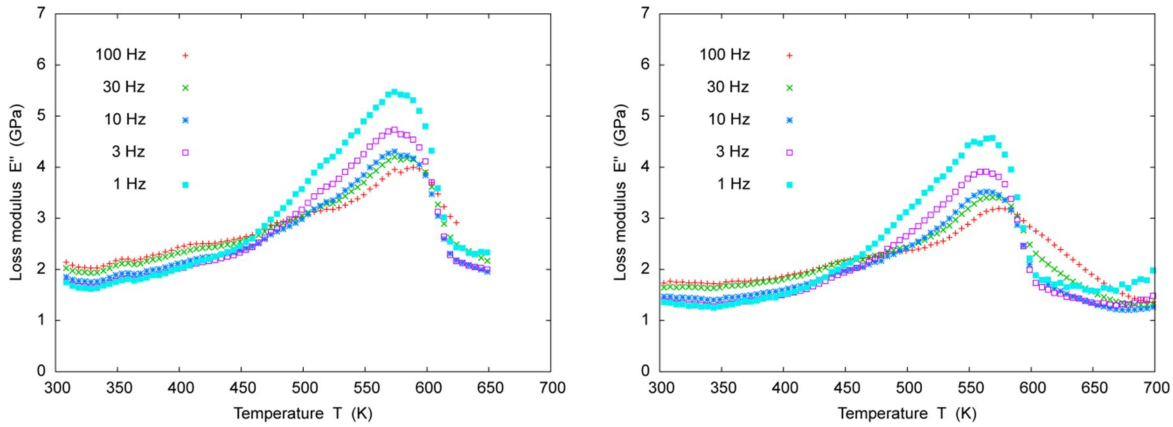
Figure 2(a) shows E'' for pure Al in the H24 temper, from RT to 648 K, while Figure 2(b) shows E'' for pure Al, from RT to 723 K, in both cases as obtained from DMA tests at frequencies ranging from 1 to 100 Hz. In this case, the behavior of E'' for pure Al shows noticeable differences to that observed for AA 7075-T6 and 2024-T3 [5,6] and AA 6082 [30]. At low temperatures, the slopes of E'' are similar for all the studied frequencies and not very steep (all show almost a plateau). At 393–533 K the slopes increase sharply. This variation in the slope is shifted towards higher temperatures with increasing loading frequency. The observed behavior may be due to the viscous loss at higher frequencies competing with shorter relaxation times. Since the relaxation time decreases with temperature due to the Arrhenius-type behavior of the relaxation rate [7], this means that the temperature above which the viscous effect exceeds the relaxation is higher for higher frequency. In other words, higher frequency viscous loss curves rise at a higher temperature than lower frequency curves.

For AA 7075-T6, 2024-T3 and 6082, the sharp growth in E'' with temperature reaches very high values without showing a peak, which is usually explained by presence of coupled relaxations [7]. On the contrary, for pure Al, E'' clearly exhibits a peak, which is achieved virtually at the same temperature for all the frequencies (around 573 K). The peak is larger (both in width and height) as the loading frequency decreases. Previous works suggest that AlZnMg alloys, AlCuMg alloys and pure Al exhibit mechanical relaxation peaks associated to dislocations and to grain boundaries [32,33]. For example, dislocation motions explain some internal friction peaks associated to semi-coherent precipitates for the alloys [34], and also the Bordoni peak, which has been extensively studied in cold-worked pure Al [7]. In this case, the observed peak corresponds to a typical internal friction peak in polycrystalline Al, related to grain boundaries [7]. In particular, the mechanism governing this relaxation is based on sliding at boundaries between adjacent grains. Upon application of stress, this process starts with the sliding of a grain over the adjacent one, caused by the shear stress acting initially across their mutual boundary. As a consequence, the shear stress is reduced gradually and opposing stresses build up at the end of the boundary and into other adjacent grains. The process terminates when the shear stress has vanished across most of the boundary, and most of the total shearing force is sustained by the grain corners.

In addition, in Figure 2, a transition is observed around 473–513 K between the low temperature region where E'' is smaller for lower loading frequencies, and the high temperature region where

E'' is smaller for higher frequencies (a stiffer response in this case is expected). Finally, after the aforementioned peak, E'' seems to increase again in Figure 2(b), particularly for the lower frequencies.

As usual, the loss tangent obtained from the DMA tests exhibits qualitatively the same behavior as E'' (the plots of loss tangent vs. temperature are included as supplementary material). The only remarkable comment is that there are no appreciable differences between the mechanical damping behavior for pure Al in the H24 temper and for pure Al, like for the E'' behavior (differences in the measured absolute values fall virtually within the instrument accuracy).



(**a**) (**b**)

Figure 2. Loss modulus E'' vs. temperature *T* from dynamic-mechanical analyzer tests at frequencies ranging from 1 to 100 Hz: (a) for pure Al in the H24 temper, from room temperature (RT) to 648 K; (b) for pure Al, from RT to 723 K.

3.3. Temperature dependence of the storage modulus

In absence of microstructural transformations, e.g., for pure Al and the alloys in the range RT–373 K, E' decreases linearly with temperature. The reasons supporting this assumption are explained herein. There is abundant literature reporting a decrease with temperature of the elastic stiffness constants of metals, e.g., the static elastic modulus of pure Al and Al alloys [35–38]. This decrease can be assumed linear in a wide temperature range. Significant deviations from linearity are only observed close to 0 K and well above the temperature range of most interest for this research, i.e., well above RT–373 K, as explained in Section 4.3. Wolfenden and Wolla [39] observed also a highly quasi-linear decrease of the dynamic elastic modulus with temperature, measured at high frequency (80 kHz) from RT to 748 K, for pure Al and for AA 6061 reinforced with alumina.

Figure 3 in [6] shows a comparison of static and high frequency dynamic elastic modulus data available in the literature with data obtained with the DMA for AA 2024-T3 and for pure Al at 1 Hz. As expected, our DMA results for pure Al in the range RT–373 K fall between the static and high frequency dynamic values of elastic modulus in the literature, but they show a pronounced decrease of dynamic elastic modulus starting around 423–523 K. This is coincident with the observed E'' peak, as shown in Figure 2. Wolfenden and Wolla [39] assumed a linear behavior from RT to 748 K and explained this in terms of the Granato-Lücke theory for dislocation damping [40], but their data are too scattered to be explained with a single mechanism in the considered temperature range, as shown by our DMA results. Moreover, the low frequency dynamic elastic modulus of pure Al should also be sensitive to micrometric mechanisms such as boundary migration during recrystallization and grain growth, as shown by Zhang et al. [41]. This mechanism is likely to be much more relevant in fatigue processes. However, the microstructural evolution and mechanical properties of AA 7075-T6 and 2024-T3 are controlled by the successive redissolution and precipitation of minority phases, and that is why it is likely that boundary migration is not the single most significant mechanism governing the behavior of their dynamic elastic moduli.

Consequently, it can be assumed that, in absence of microstructural transformations, E' decreases linearly with temperature. Accordingly, Table 1 shows the slope of the decrease of E' with temperature for pure Al in the H24 temper and for pure Al, as obtained from linear regression of the DMA experimental results. In the following, to refer properly to this parameter on the basis of its physical meaning, it is termed "temperature softening coefficient", E'_0. Finally, it is important to note that the larger decrease of E' for low frequencies is typically explained by the Arrhenius-type behavior of the relaxation rate. That is, the mechanical relaxation time decreases with temperature [7] so that, at low frequencies, the shorter relaxation times lead to responses with larger values of E'' and smaller values of E'.

Table 1. Temperature softening coefficient E'_0, for pure Al in the H24 temper and for pure Al, as obtained from linear regression of dynamic-mechanical analyzer test data.

loading frequency [Hz]	E'_0 (pure Al in H24 temper) [MPa·K^{-1}]	E'_0 (pure Al) [MPa·K^{-1}]
100	-23.1	-26.4
30	-25.24	-27.7
10	-26.4	-29.1
3	-27.9	-31.0
1	-29.7	-32.2

3.4. Effect of internal friction on fatigue strength

Considering that viscoelasticity is linked to the fatigue and yield stress behavior [7], it is likely that the decrease of fatigue and yield stress behavior observed in metals as temperature increases is associated to the increase of internal friction. The reasons supporting this assumption are explained herein. Namely, Zhu et al. [14] observed that the fatigue strength of an Al alloy decreases with temperature, with a behavior that follows closely the temperature dependence of the yield and tensile strength. Also, Liaw et al. [16] suggested that the fatigue limits and fatigue lives of steels decrease due to the temperature increase caused by plastic deformation. Finally, from the work by Amiri and Khonsari [4], it appears that the more intense the hysteresis heating of a metal during dynamic loading, the lower its fatigue resistance. Extrapolating the assumption stated above to AA 7075-T6, 2024-T3 and AA 6082, their yield and fatigue strength would decrease with temperature according to the observed increase in internal friction in the tested temperature range, as shown in [5,6] and [30], respectively. For pure Al, the yield and fatigue strength would decrease up to the E'' peak.

Furthermore, if considering the influence of loading frequency on internal friction, our results suggest that below 423 K the yield and fatigue strength would decrease with increasing frequency, while at high temperatures these properties would increase. These presumptions agree with some of the research reported in the literature, but still there is controversy about the effect of frequency on the fatigue response, as shown in Section 1.1. For example, our findings agree with results from low frequency investigations for Al alloy RR58 at 423 K [25] and for AA 2650-T6 at 175°C [26], pointing that fatigue life is sensitive to frequency and that it increases with loading frequency. There is agreement also with results for E319 cast Al alloy at 423 and 523 K, pointing that fatigue life at 20 kHz was longer than at 75 Hz [14]. However, this behavior is attributed to environmental effects on FCG rate, rather than intrinsic temperature or frequency effects.

3.5. Onset frequency of fatigue effects

It is reasonable to accept that, at sufficiently low loading frequency, fatigue effects become negligible. Indeed, in a series of dynamic loading tests with loading frequency decreasing to very low values, the test time scale will eventually become much larger than the largest mechanical relaxation time for any of the possible relaxation processes for the tested material. That is, the reduction in the loading frequency is equivalent to an increase in the reaction time and, therefore, there is a threshold frequency below which the reaction time is longer than the relaxation time. Consequently, the viscous effect as the loss mechanism is negligible as compared with the relaxation. This means that eventually E" will decrease to a level that there will be no appreciable frictional energy loss due to relaxation effects. Recalling the statement by Amiri and Khonsari [4] that some degree of hysteresis heating (which is caused by the energy dissipated due to internal friction) is necessary for metals to experience fatigue, the conclusion is that, eventually, fatigue effects vanish. This hypothesis is in line with research by Henaff et al. [26] and Nikbin and Radon [25], suggesting the existence of a critical value of loading frequency below which, for a given temperature, the material exhibits a static-like response (relaxed case), crack growth is sensitive to frequency and static creep dominates instead of pure fatigue mechanisms.

Next, a procedure to estimate this threshold frequency is proposed based on the frequency dependence of the temperature softening coefficient E'_0. The procedure is illustrated in Figure 3, which shows E'_0 as a function of the loading frequency for pure Al in the H24 temper and pure Al, as obtained from linear regression of DMA data, and for AA 7075-T6 and 2024-T3, as obtained in [5,6]. These values of E'_0 are compared to average values of the rate of loss of static elastic modulus with temperature, as obtained by linear regression of data in the literature for pure Al [36] and AA 2024 [35]. To calculate these averages, data from Kamm and Alers [37] and from Varshni [38] were disregarded as they correspond to temperatures below RT. Data from Wolfenden and Wolla [39] were also disregarded since these data are too scattered in a broad temperature range, and thus the computable slope is probably not be representative.

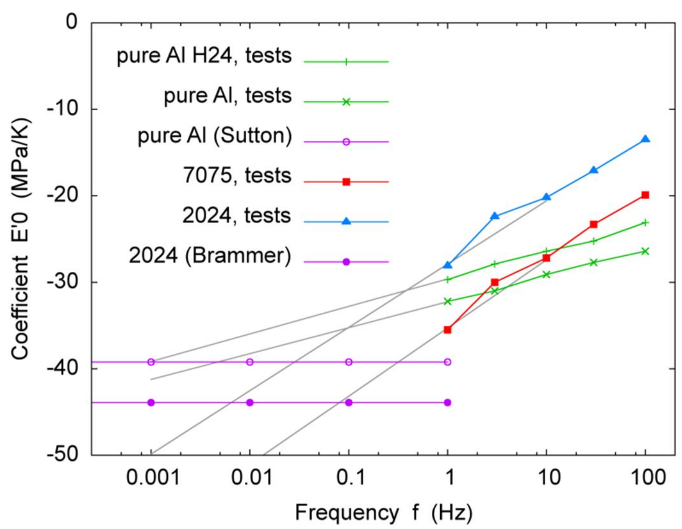


Figure 3. Temperature softening coefficient E'_0 vs. frequency *f* for pure Al in the H24 temper, pure Al, Al alloy (AA) 7075-T6 and AA 2024-T3, as obtained from linear regression of test data from the dynamic-mechanical analyzer. Logarithmic tendency lines fitted to the data to extrapolate it to lower frequencies are also shown, as well as the rates of loss of static elastic modulus with temperature, as obtained by linear regression of data in the literature for pure Al [36] and AA 2024 [35].

Assuming that, at the threshold frequency, E'_0 should become equal to the rate of loss of elastic modulus with temperature under static loading conditions, this threshold frequency may be estimated by intersection of the latter rate with a tendency line extrapolating the behavior of E'_0 measured experimentally to lower frequencies. In the example shown in Figure 3, the intersection of a logarithmic tendency line with the rate of loss of static elastic modulus for pure Al in the H24 temper and pure Al gives a threshold frequency of around 0.001 and 0.005 Hz, respectively. For AA 2024-T3, the threshold frequency obtained with the same procedure would be about 0.006 Hz. For AA 7075-T6, no data on the variation of the elastic stiffness constants with temperature was found in the literature, but using the data available for AA 2024 and pure Al, a threshold frequency of 0.075 and 0.350 Hz would be obtained, respectively. These results are similar to those in the literature: Henaff et al. [26] reported a critical frequency of 0.020 Hz for AA 2650-T6, and Nikbin and Radon [25] reported that the transition region is 0.100 to 1 Hz for cast Al alloy RR58 at 423 K. However, to better assess the performance of the proposed procedure, further comparison with experimental data on fatigue response at very low frequencies is necessary. Unfortunately, there is a lack of this type of test data [26], due to the extremely long testing time. It is interesting to note that, on one side, according to our results, fatigue effects would seem to appear at lower loading frequencies for pure Al, compared to the alloys. Thus, apparently the precipitation structure in the alloys would cause not only hardening but would also enable for the alloys to be loaded at a wider range of low frequencies without experiencing fatigue. On the other side, fatigue would appear already at lower frequencies for pure Al in the H24 temper compared to pure Al. In this case, the reason may be the lower ductility (and thus lower resistance to FCG) associated to the H24 temper.

The proposed procedure for the determination of the threshold frequency is a major result of this work. Low frequency fatigue experiments are, by definition, very long. Furthermore, there is still controversy about the effects of the exposure to the environment on the experimental results, as it is not feasible to perform longstanding experiments in constant environment conditions. Besides, the environment during service life is likely to be different than that during the experiments, and thus estimations of the threshold frequency based on conventional, time-consuming fatigue experiments are likely to be inaccurate. The determination of the threshold frequency from the data obtained with the relatively short DMA tests (less than 3h) reflects intrinsic properties of the material only. This offers also a standardized method, which allows precise comparison between different alloys. Furthermore, it is quite insensitive to the specific instrumental range available for the tests, provided the range is large enough to allow a consistent regression fit.

4. Conclusions

It was suggested that some degree of hysteresis heating is necessary for metals to experience fatigue when subjected to dynamic loading. Hysteresis heating is caused by energy dissipated due to internal friction, and thus an increase in the latter should have an effect on the fatigue response. In particular, the results of this research suggest that the decrease of yield and fatigue behavior reported for Al alloys as temperature increases may be associated to the increase of internal friction with temperature. Due to the Arrhenius-type behavior of relaxation processes, the relaxation time decreases with temperature. The reduction in the loading frequency is equivalent to an increase in the reaction time and, therefore, there is a threshold frequency below which the reaction time is longer than the relaxation time. Consequently, the viscous effect as the loss mechanism is negligible as compared with the relaxation. In other words, with the dynamic loading frequency decreasing to very low values, eventually there will be no appreciable frictional energy dissipation, and thus no hysteresis heating, due to mechanical relaxation phenomena. Thus, below the threshold frequency the material will exhibit a static-like response (relaxed case), such that creep mechanisms dominate and fatigue effects are negligible. In this work, an alternative procedure to the time-consuming conventional fatigue tests is proposed to estimate this threshold frequency, based on the frequency dependence of the slope of the initial decrease of E' with temperature, which in this work is termed temperature softening coefficient. The interesting point of our approach comes from the fact that this coefficient is easily obtained from the relatively short DMA tests, hence allowing for a fast estimation of the threshold frequency. For pure Al, AA 2024-T3 and AA 7075-T6, the threshold frequencies obtained with this procedure are 0.001–0.005, 0.006 and 0.075–0.350 Hz, respectively. This suggests that fatigue effects start to appear at lower loading frequencies for pure Al, while the alloys may be loaded at a wider range of low frequencies without experiencing fatigue, probably due to effects related to presence of precipitates. However, to better assess the performance of the proposed procedure, further comparison with experimental data on fatigue response at very low frequencies is necessary.

Supplementary Materials: The following supplementary material is available online at www.mdpi.com/link: 1) Spreadsheet S1 - DMA test data (MS Excel file), which includes the measured values of storage modulus E', loss modulus E'', and loss tangent (also termed mechanical damping or $\tan\delta$) for pure Al in the H24 temper, pure Al, AA 7075-T6 and AA 2024-T3; 2) Figure S2a - mechanical damping of pure Al in the H24 temper and Figure S2b - mechanical damping of pure Al.

Acknowledgments: Work supported by the MINECO grant FIS2014-54734-P and the Generalitat de Catalunya/AGAUR grant 2014 SGR 00581. As part of these grants, we received funds for covering the costs to publish in open access.

Author Contributions: J.I.R. conceived, designed and performed the experiments; J.I.R. and D.C. analyzed the data and wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

Abbreviations

The following abbreviations are used in this manuscript:

AA: Aluminum alloy(s)
bcc: Body-centered cubic
CFCG: Creep-fatigue crack growth
CCG: Creep crack growth
DMA: Dynamic-mechanical analyzer
fcc: Face-centered cubic
FCG: Fatigue crack growth
GPBZ: Guinier-Preston-Bagariastkij zones
GPZ: Guinier-Preston zones
HCF: High cycle fatigue
RT: Room temperature
VHCF: Very high cycle fatigue

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