

## THERMAL AGING EFFECT ON MECHANICAL PROPERTIES OF 30% GLASS FIBER REINFORCED POLYAMIDE 11

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**ABSTRACT:** This study investigates the effects of artificial thermal aging on the mechanical properties of 30% glass fiber-reinforced Polyamide 11 (PA11-GF30). Tensile and compression tests were conducted on specimens aged at 125°C, 150°C, and 175°C for up to 4152 hours. The results indicate a progressive decline in mechanical properties, with tensile strength decreasing from an initial 104.6 MPa to 92.63 MPa (12.4% reduction) at 125°C, 73.96 MPa (34.3% reduction) at 150°C, and 74.36 MPa (33.8% reduction) at 175°C. Similarly, compressive strength declined from 135.59 MPa to 122.81 MPa (9.4% reduction) at 125°C, 124.28 MPa (8.3% reduction) at 150°C, and 104.76 MPa (22.7% reduction) at 175°C. Notably, an initial strengthening phase was observed due to post-crystallization, particularly at lower temperatures. Elongation at break exhibited a sharp decline, confirming the transition from ductile to brittle behavior, with samples at 150°C and 175°C reaching the 5% brittleness threshold after 2016 hours and 1344 hours, respectively. These findings provide critical insights for the design and application of PA11-GF30 in high-temperature environments, supporting its use in automotive, aerospace, and industrial sectors requiring durable polymer-based components.

**KEY WORDS:** thermal aging; mechanical properties; polyamides; PA11.

### 1 INTRODUCTION

Thermal aging refers to the degradation of polymers due to exposure to elevated temperatures, which can occur during processing, storage, or in-service conditions [1]. The effects of thermal aging on PA11 encompass a broad range of physical and chemical transformations, including changes in mechanical properties, molecular structure, morphology, and surface characteristics [2]. Understanding thermal aging is crucial for predicting the long-term performance and durability of polymer-based products in real-world applications.

Polyamide 11 (PA11), a thermoplastic polymer derived from renewable sources, has garnered significant attention in various industrial applications due to its ex-

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ceptional mechanical properties, chemical resistance, and environmental sustainability [3]. PA11 is used in a large number of applications due to its easy processing, high and low temperature performance ( $-40^{\circ}\text{C}$  /  $+130^{\circ}\text{C}$ ), high dimensional stability, and low density. Many industries around the world (e.g. automotive, aerospace, textile, oil & gas, electronics) have used PA11 for many decades for its long-term durability.

The PA11 under study was Rilsan®BZM 30 O TL (PA11-GF30) from Arkema S.A. The material is a 30% fiberglass reinforced polyamide 11 produced from castor oil. Arkema S.A. specifications state this natural grade is designed for injection molding and the percentage of renewable carbon is  $>97\%$  according to ASTM 6866 [4].

The PA11 matrix accommodates many additives and filling agents, such as plasticizers, stabilizers, colorants, lubricants, impact modifiers, etc [5].

However, like many plastics, PA11 is subject to aging phenomena over time, which can compromise its performance and reliability in service [6]. Among the various aging mechanisms, thermal aging stands out as a prominent factor influencing the long-term behavior of PA11. The current experiment involves dry thermal aging in the absence of any additional environmental factors such as moisture or chemicals. There is no load applied to the specimens during the aging process.

The paper aims to examine aging phenomena in PA11 by subjecting samples to carefully controlled thermal aging conditions. Through mechanical testing, this research endeavors to elucidate the complex relationship among temperature, time, and material properties degradation throughout the process of dry thermal aging. The relationship between aging and alterations in the mechanical characteristics of PA11 is not thoroughly documented or comprehensively understood. Ultimately, this study contributes to advancing the utilization of PA11 as a sustainable and high-performance polymer in diverse industrial applications.

## 2 THERMAL AGING EXPERIMENT

### 2.1 SPECIMEN PREPARATION FOR TENSILE AND COMPRESSION TESTS

The samples used for the thermal aging experiment were produced by injection molding using the Arburg 270 M molding machine shown in Fig. 1.

Table 1 shows the injection molding machine parameters used for manufacturing the specimens. A special injection mold tool has been designed for manufacturing multipurpose test specimens according to the requirements specified in the standard EN ISO 20753:2024. The mold is specialized in injection molding of multipurpose test specimens type A1 and its design corresponds to the standard EN ISO 294-1:2017 [7]. The specimens can be directly used for tensile tests according to EN ISO 527-2.

For compression tests the middle section of the dog bone specimen was cut into five pieces on a CNC machine to the dimension specified at EN ISO 604:2002.



Fig. 1. Multipurpose specimens manufacturing from PA11 resin.

Table 1. Key Injection Molding Parameters used for manufacturing specimens from PA11 with 30% Glass Fibers

Parameter	Value	Parameter	Value
Injection Pressure	120 Mpa	Injection Speed	150 mm/s
Melt Temperature	250°	Mold Temperature	80°
Clamping Force	13 kN	Holding Pressure	25 Mpa
Cycle Time	35 s		

## 2.2 ACCELERATED AGING

The samples were placed inside three different drying ovens, Horyzont SPT 200, which can maintain stable temperature over time throughout the aging process. The three different temperatures of 125°C, 150°C, and 175°C were selected for the artificial aging of PA11 in drying ovens based on the guidelines provided by IEC 60216 [8]. This standard establishes accelerated aging protocols to determine the thermal endurance of insulating materials. The chosen temperatures represent a range that accelerates the aging process while remaining relevant to the expected operating conditions of PA11. By using these temperatures, it is possible to extrapolate the material's long-term performance and degradation behavior under typical service conditions, ensuring reliable and reproducible results.



Fig. 2. Arrangement of specimens into drying oven.

The samples are arranged within the aging oven in a manner that ensures equal exposure to the temperature conditions as shown in Fig. 2.

Each specimen is positioned so that it does not touch the others, allowing uniform exposure to the oven temperature and preventing any shadowed areas on individual specimens.

A predetermined period of at least 4000 hours of aging is selected for the three different temperatures. A detailed schedule was created wherein the specimens should be removed from the oven and allowed to cool down to room temperature. At higher temperatures the specimens were taken more often from the ovens than at lower aging temperature since it was assumed that the aging process would happen faster. Later on, all of those specimens were tested for tensile and compression properties.

### 3 MECHANICAL ANALYSIS (ISO 527, ISO 604)

#### 3.1 TENSILE STRENGTH – ISO 527

Rilsan®PA11 has high elongation at break and high tensile strength at break and at yield. It is one of the toughest high performance polymers and is therefore used extensively in demanding applications [5].

The tensile test according to ISO 527 is a most used method for evaluating the mechanical properties of plastics providing essential data for engineering design and material specifications [9].

Experiments were made on Instron 1185 tensile testing machine. A preload was applied to the specimens to prevent slipping or shifting in the machine jaws during loading. The specimens were tested with a displacement rate of 5 mm/min until fracture.



Fig. 3. Samples of PA11 exposed to accelerated aging at 125°.

The stress for each specimen was calculated based on its initial cross-section. The elongation was calculated based on the initial length of the specimens and the tensile machine displacement [10].

The tensile strength was estimated by dividing the maximum load in each stress-strain curve by the original cross-sectional areas.

### 3.2 COMPRESSION STRENGTH – ISO 604

The compression properties characterize the strength, deformation and stiffness behavior of plastics under quasi-static uniaxial compression load conditions. The commonly used standard for the compression test of plastics is the ISO 604 (2002): Plastics – Determination of compressive properties. The compression specimens are manufactured from the multipurpose ones and have dimensions of  $10 \times 10 \times 4$  mm.

Compression experiments were made on the same testing machine at displacement rate of 1 mm/min.

For each aging period five samples were tested. The mean and standard deviation were calculated and are depicted in the graphs below.

## 4 RESULTS AND DISCUSSION

### 4.1 VISUAL CHANGE OF SPECIMENS

The initial visible change observed after accelerated dry aging was the color shift (yellowing) in PA11 shown in Fig. 3.

This phenomenon has been already reported in studies dealing with thermo-oxidative degradation and photo-degradation [10–12]. According to literature data the yellowing of polyamide materials is mainly caused by the formation of pyrrole derivatives [10–12] or conjugated enamine or enone groups [11, 13]. Due to the degradation process, tested samples move from uncolored ones to yellow and finally turn to black

as reported by Rudzinski et al. [11]. After 4000 hours the specimens became black at 125°C. At 150°C and 175°C the specimens became black after the first 500 hours.

#### 4.2 MECHANICAL BEHAVIOR – TENSILE PROPERTIES

The typical stress-strain curves of plastics, as described in the standard ISO 527 [9], reveal distinct behaviors depending on the material's state and environmental conditions. For unaged specimens, PA11 exhibited a ductile response, reaching a maximum stress value before failure. In this case the material undergoes high strain before breaking.

Upon aging, PA11's tensile behavior transitioned towards brittleness, as evidenced by a marked reduction in strain at break and a shift to behavior represented as brittle material by the standard. This shift is characterized by the specimen fracturing at its maximum tensile stress, indicating that the tensile strength coincides with the stress at break. The degradation of tensile properties over time is attributed to molecular-level changes, such as chain scission and oxidative reactions, which compromise the polymer's ductility [10, 11].

The stress-strain results analysis revealed significant aging-dependent variations across the three test temperatures shown in Fig. 4. The tensile strength of PA11 decreased over the aging period, with distinct trends observed for each temperature.

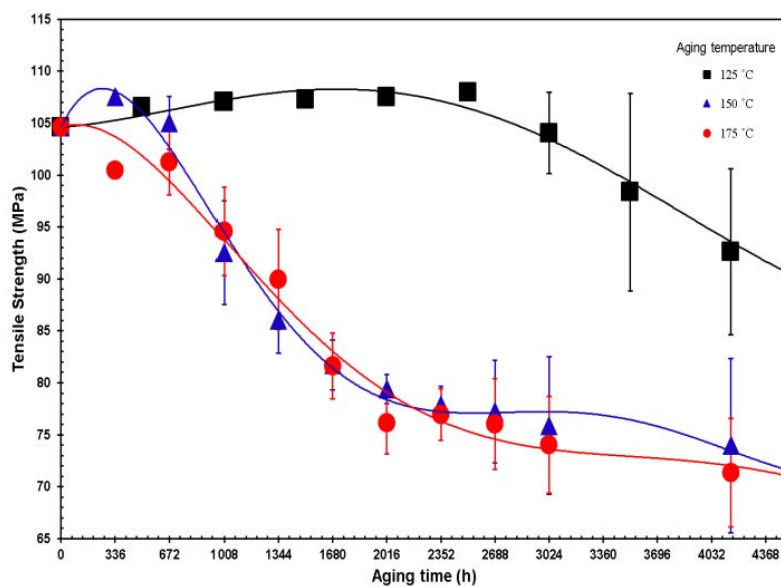


Fig. 4. Tensile strength over aging time.

Unaged specimens started with an initial tensile strength of 104.6 MPa, closely aligning with manufacturer specifications of 117 MPa [4].

At 125°C, PA11 initially exhibited a minor strengthening phase, reaching 3.34 MPa above the baseline tensile strength after 2520 hours. This phenomenon is likely due to post-crystallization processes, wherein polymer chains reorganize into more stable crystalline structures during thermal exposure. However, extended aging led to a degradation phase, with the tensile strength dropping to 92.63 MPa at 4152 hours, representing a 12.4% reduction.

Similarly, at 150°C, a transient strengthening phase was observed within the first 336 hours, followed by a more pronounced degradation phase, culminating in a 34.3% reduction in tensile strength (to 73.96 MPa) at 4152 hours. At 175°C, the strengthening phase likely occurred between testing intervals and was not directly observed. By 4152 hours, the tensile strength had decreased to 74.36 MPa, reflecting a 33.8% reduction. These results highlight the accelerated degradation effect of higher temperatures, consistent with the Arrhenius relationship that predicts faster reaction rates at elevated temperatures [7, 10, 13].

Notably, after 3000–4000 hours, the degradation curves for both temperatures 150°C and 175°C become almost asymptotic, suggesting that tensile strength reduction plateaus over time. This indicates that no significant further decrease in mechan-

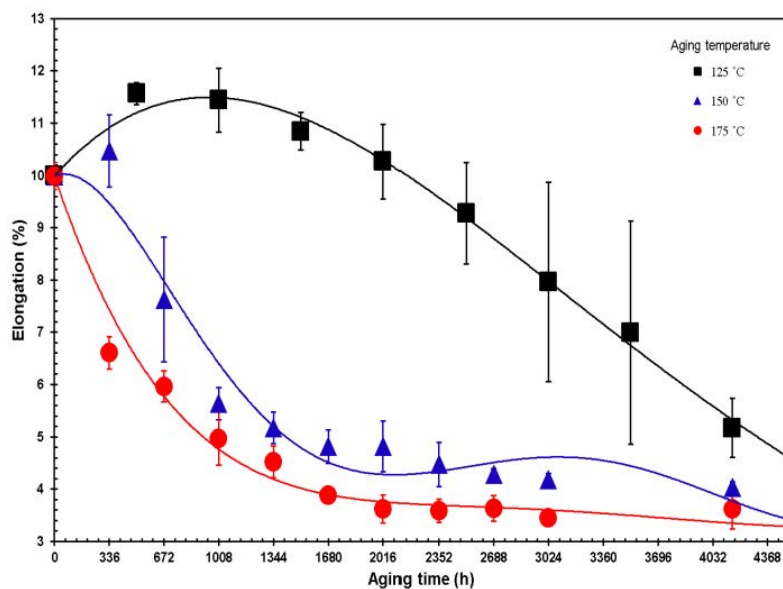


Fig. 5. Elongation at break.

ical properties is expected beyond this exposure period.

The strain behavior further supports the brittleness transition in aged PA11. As seen in Figure 5, the material crossed the 5% elongation at break threshold, typically defining the brittle material regime. At 125°C, this threshold was reached at 4152 hours, while at 150°C and 175°C, it occurred significantly earlier, at 2016 hours and 1344 hours, respectively. The early onset of brittleness at higher temperatures underscores the temperature-dependent nature of aging in PA11 [11, 13].

#### 4.3 MECHANICAL BEHAVIOR – COMPRESSION PROPERTIES

The compressive properties of PA11 were analyzed following the ISO 604 standard, which defines methods for determining the compressive strength, deformation, and stiffness behavior of plastics under quasi-static uniaxial loading [11]. This study provides critical insights into how thermal aging affects PA11's resistance to compressive stresses, complementing the tensile behavior analysis.

Compression testing revealed an initial compressive strength of 135.59 MPa for unaged specimens. This value is 22.8% higher than the tensile strength of the same material, highlighting the intrinsic resistance of PA11 to compressive forces compared to tensile stresses. Similar to the trends observed in tensile test results, the compressive strength of PA11 decreased over time with thermal aging (Figure 6), reflecting the molecular degradation mechanisms [11, 12].

At 125°C, the compressive strength initially exhibited a minor increase, reaching 138.11 MPa after 2520 hours. This strengthening phase can be attributed to post-crystallization, where molecular chains reorganize into more stable configurations, temporarily enhancing mechanical performance. However, by 4152 hours, the compressive strength had decreased to 122.81 MPa, marking a 9.4% reduction from the initial value.

At 150°C, the material displayed two distinct strengthening zones: between 336 and 1680 hours, and again between 1680 and 2016 hours. These intermittent strengthening phases are consistent with post-crystallization dynamics and localized relaxation of internal stresses induced by thermal exposure. Despite these effects, extended aging led to a compressive strength reduction to 124.28 MPa at 4152 hours, reflecting an overall decrease of 8.3%.

In contrast, at 175°C, the compressive strength steadily declined throughout the aging process without any observable strengthening phases. By 4152 hours, the compressive strength had dropped significantly to 104.76 MPa, representing a 22.7% reduction. This rapid degradation aligns with the accelerated aging effects of higher temperatures, wherein the rate of molecular degradation surpasses any temporary gains from post-crystallization [11, 13].

The strain behavior during compression further underscores the impact of aging

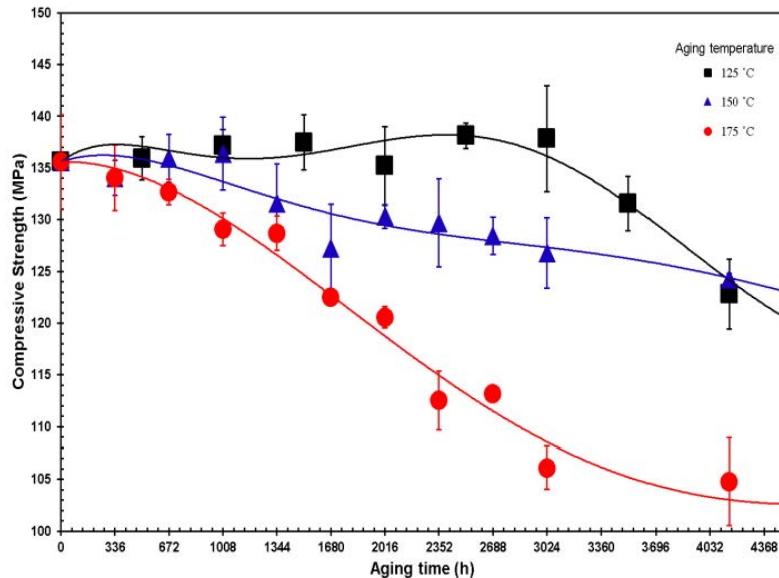


Fig. 6. Compressive strength.

on PA11's structural integrity. As the material aged, compressive strain decreased from 10% of unaged specimens to 7% after 4368 hours, reflecting reduced ductility and increased brittleness.

## 5 CONCLUSIONS

This study examined the thermal aging effects on the mechanical properties of Polyamide 11 (PA11) reinforced with 30% glass fibers under controlled dry accelerated aging conditions. Experimental results show that PA11 undergoes gradual degradation in tensile and compressive strength over time, with the rate of decline strongly influenced by temperature. Despite this, the material exhibited remarkable performance during initial exposure, with moderate temperatures (125°C and 150°C) inducing a temporary strengthening phase attributed to post-crystallization processes.

The findings highlight the suitability of PA11 for applications at elevated temperatures, particularly in industries requiring lightweight mechanically robust components. The material's low density and high strength make it an attractive option for advanced applications, including injection-molded parts used in automotive, aerospace and electronics sectors. Additionally, the demonstrated resilience to compressive loads further broadens its applicability in structural components subjected to such stresses.

This research contributes to the scientific understanding of PA11's aging behav-

ior, offering engineers valuable insights for designing durable components for high-temperature environments. The results also support the broader adoption of PA11 in high-performance applications, leveraging its exceptional mechanical properties and environmental sustainability.

To further validate the conclusions drawn in Section 4.1, future research will include Thermogravimetric Analysis (TGA) of PA11 reinforced with 30% glass fibers. This analysis will provide deeper insight into the thermal stability and degradation mechanisms of the material by quantifying weight loss as a function of temperature. TGA results will complement the existing mechanical data and help establish a more comprehensive understanding of the material's long-term behavior under thermal aging conditions.

Additionally, the observed thermal aging trends in PA11 lay the groundwork for future research on predicting mechanical degradation using temperature-based models similar to the Arrhenius equation. Applying this approach could help quantify long-term performance changes under various thermal conditions. Future studies should focus on validating these models and expanding predictions to a wider range of environments.

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