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Impact of post-curing duration on mechanical, thermal and tribological behavior of an organic friction material

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ABSTRACT

This paper aimed to study the influence of post curing duration on mechanical, thermal and tribological behavior of friction materials elaborated with simplified formulation. Surface mechanical properties and thermal conductivity were analyzed and tribological behavior was studied for various thermal severities of sliding conditions. Results indicated that post curing for long duration allowed to reduce thermal conductivity and to homogenize the surface mechanical properties of the friction material. Concerning tribological behavior, it was shown that a longer post curing duration permitted to reduce the level of friction and to increase wear resistance. Worn surface morphology investigation using SEM revealed that wear and friction mechanisms involved in the contact were sensitive to post curing duration.

1. Introduction

Brake manufacturers have to meet a number of comprehensive tribological and economic requirements demanded by their customers, particularly, acceptable friction level, friction stability, wear resistance irrespective of brake conditions and cost reduction. Besides composition of the friction material plays a major role in satisfying the target properties [1,2], manufacturing process (mixing, preforming, hot molding and post curing) as well as the third body formed at the interface during braking, affect, also, significantly the friction performance [3,4]. Some researchers [5] proved that the manufacturing process parameters depend strongly on the complex formulation of friction materials.

Post curing is a crucial step in defining the final properties of friction material. Indeed, it was considered as an additional curing, when the cross linking phase of the resin was not achieved during hot molding step [6]. In this case, it was reported that post curing step enhanced the thermosetting polymer (such as phenolic resin) resistance at high temperature which is dependent on the thermal stability of the material after cross linking [7]. Some studies treated the post curing step as an annealing treatment which allowing relieving the residual stresses introduced during matrix polymerization and during cooling after hot molding step [8]. It was shown that annealing treatment had significant effects on the mechanical performance of the composite material. It permitted to reduce its stiffness and strength [9]. Others studies established that post curing could contribute to modify the mechanical properties of friction materials, regardless of their microstructures [10]. Among post curing parameters, few researchers shed light on duration to understand its impact on tribological properties, particularly friction coefficient and wear resistance. Aleksendric and Senatore [5] indicated how wear of the friction material can be sensitive versus post curing duration without giving an accurate link between wear and post curing duration. In varying post curing duration from 10 h to 16 h, Ertan and Yavuz [11] claim that the resin can be decomposed if the material is maintained for a long post curing duration at high temperatures, which may give rise to wear resistance decrease and friction coefficient reduction.

Concerning third body, it was established that the rubbed friction material surface can be divided in two different contact zones: a first contact zone defined by the polymeric matrix contact and a second contact zone defined by hard patches or flat plates [12]. Such plates were composed of constituents from degraded friction material and from damaged disc. The formation, stability and durability of the third body flat plates had a significant effect on brake performance and wear behavior of organic friction materials [13–16]. The thickness and surface morphology of the flat plates were highly

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dependent on sliding conditions such as temperature, sliding speed, applied pressure and on chemical state of the friction material constituents in the flat plates [17,18]. According to Sellami et al. [19], friction performance was found to be independent on flat plates thickness but sensitive to their composition. Researchers have shown that increasing sliding speed or applied pressure can promote trapping third body and flat plates coalescence [20] leading to wear increase [21,22]. The combined effect of pressure and sliding speed on wear is more significant [23]. The third body layer formed can act as a protective layer that reduces the friction level [21]. Contrariwise, other researchers have shown that flat plates formation can increase the coefficient of friction [24].

Previous research showed the importance of organic, fibrous constituents, and solid lubricants in supporting flat plates at the friction interface [25], leading to large load-bearing flat plates [26].

Understanding the relation between friction materials performances and manufacturing process is crucial to improve the efficiency of braking. Though literature, studies reporting the relationships between post-curing duration and tribological performance of the friction materials are very few. Also studies involving the link between post curing duration and thermal and mechanical properties as well as friction and wear mechanisms are yet unexplored. In the current work, an effort has been made to bridge this gap by studying mechanical, thermal (thermal conductivity) and tribological properties (friction and wear) and thereby establishing a correlation about such properties of two friction materials post cured for two different duration.

2. Experimental work

2.1. Friction materials preparation

Friction materials designed for this work had a simplified formulation derived from a complex industrial one containing 13 constituents [27], aiming to limit synergistic effects between constituents as well as between formulation and process. Formulation constituents were classified to six categories according to their main role in the mechanical and tribological behavior of the brake friction material: binder, fibers, fillers, abrasives, lubricants and friction modifiers. So, to develop the simplified formulation, selection of constituents was based on their weight percent values in the formulation categories. The weight percent of each constituent corresponded to the weight percent of each category. For example, the filler category of the industrial formulation contained barite, calcium carbonate and chalk which represented 45%. In simplified formulation, we selected barite that present the higher W% and we attributed for it the weight percent of filler category (45%). Therefore, simplified formulation contained only six different constituents (Table 1).

The industrial process consisted in dry-mixing of the preweighed constituents, pre-forming at 20 MPa, hot-molding at 140 °C during 18 min at 20 MPa and finally post curing at 160 °C during 10 h. At the end of hot molding, friction material had a plate shape of size 400 × 400 × 16 mm^3. As, hot molding was accompanied by degassing by the upper surface (US) of the plate, differences were expected between the two surfaces. So, they were distinguished by marking the upper surface. Nine samples of dimensions 65 × 65 × 16 mm^3 were machined from a quarter of the molded plate. Two samples were used to characterize the molded material in terms of polymerization and acetone extraction.

Table 1
Friction material formulation.

Category	Constituent	Weight %
Binder	Phenolic resin	14
Filler	Barite	45
Fiber reinforcement	Rockwool fiber	22
Abrasive	Aluminum oxide	2
Lubricant	Graphite	10
Friction modifier	Rubber	7

- Polymerization: DTA tests were performed on samples of the molded material to evaluate their polymerization. Results showed that polymerization was achieved after hot-molding step.
- Acetone extraction: it was equal to 1.2 for the molded material. This value was within the acetone extraction values range of cross linked materials [28,29]. So the molded material was cross linked before post-curing step.

Therefore, post curing could not contribute to modify chemical properties of the friction material. Thus, we were interested, in this paper, in studying the influence of post curing, particularly duration, in mechanical, thermal and tribological properties. To this end, two durations were selected: the first is 10 h which represented the industrial duration and the second was 6 h. Therefore, two materials were elaborated. Mixing, preforming and hot molding were conducted in the industrial chain, but post curing was realized in a laboratory furnace preheated at 160 °C (Fig. 1) to ensure, on one hand, the homogeneity and stability of temperature during post curing and samples reproducibility, on the other hand. To apply the post curing treatment properly, we ensured that temperatures in the furnace and in the sample were stable and homogeneous at 160 °C for a maximum duration of 10 h. A thermocouple was used to measure the temperature in the furnace chamber (denoted T–F), and it was attached to a metal plate, taking care not to cover the free end of the thermocouple to avoid any thermal resistance effect. In the sample, temperature was monitored using two thermocouples T1 and T2. These thermocouples were introduced into two holes, bored at the upper surface using a drill of 1.5 mm diameter, and spaced of 10 mm (Fig. 1b). It was shown that temperature achieved 160 °C after one hour and a half, beyond which, it remained quasi-stable until the end of post curing. In the following, the post cured materials at 160 °C for 10 and 6 h, are denoted M-P10H and M-P06H respectively.

2.2. Surface mechanical characterization

To characterize the surface mechanical behavior of both materials M-P10H and M-P06H, we performed an instrumented indentation testing using macro durometer ZHU_zwicki_Line. Test parameters were kept identical to those used by Baklouti et al. [27]. Since the tested materials presented high heterogeneity due to their complex microstructure, each surface (upper and lower) of each sample was indented in 18 points identified in Fig. 2. To guaranty measurements independency, a minimum distance between two indentation points was respected. From each charge/discharge cycle, the energy associated to each indentation point was calculated by the trapezium rule. These energy values were chosen to evaluate the impact of post curing duration on mechanical behavior. Indeed, they were processed using STATISTICA software, through LEVENE and ANOVA tests with a threshold α of 5% (or 0.05 if it is expressed as probability) to evaluate respectively the equality of variances and the equality of mean values. Indeed, before moving to ANOVA tests, it was necessary to check the equality of variance.

To do so, a null hypothesis "H0" was formulated to test the variance of data groups (indentation energy values in our case). H0 affirmed that « Data group's variances are equal ». p-LEVENE value was calculated. This value was considered as the risk that there was to reject this hypothesis: