VERS UN MATERIAU A LA RESILIENCE MECANIQUE DIMINUEE

1. Introduction

Une voie intéressante de production de biocarburants de seconde génération consiste en une gazeification de la biomasse suivie d'une synthèse Fisher-Tropsch. Cependant de fines particules (entre 100 et 200 µm) sont indispensables afin d'optimiser les réactions de synthèse, les rendements en gaz ou encore la coulabilité du matériau dans la chaîne de production (Simmons 1986; Bergman et al. 2005; Wei et al. 2006). Comme la structure hétérogène et fibreuse de la biomasse rend l'étape de broyage complexe et gourmande en énergie, une étape de prétraitement est indispensable. Dans ce domaine, la torréfaction semble être un prétraitement idéal.

La partie qui suit a pour objectif de contribuer à l'évaluation du rôle de la torréfaction sur le comportement au broyage de la biomasse grâce au dispositif expérimental précédemment décrit. Comme le bois représente environ 80% de la production de matière ligno-cellulosique dans le monde il a particulièrement été étudié dans cette partie. La diminution de résilience mécanique du pin maritime (*Pinus pinaster*) et du chêne pédonculé (*Quercus robur*) traités thermiquement a notamment été évaluée.

2. Grindability of heat treated wood assessed by a custom impact device

Floran Pierre[a], Giana Almeida[b], Patrick Perré[c*]

[a]AgroParisTech, UMR1092, LERFoB Bois Biomateriaux Biomasse team, Nancy, France;
[b]INRA, UMR1092, LERFoB Bois Biomateriaux Biomasse team, Nancy, France;
[c]ECOLE CENTRALE PARIS, LGPM, Grande Voie des Vignes, 92 295 Châtenay-Malabry, France
*Corresponding author: patrick.perre@ecp.fr

2.1. ABSTRACT

The BtL (biomass to liquid) chain required fine particles to be produced before the introduction in the gasifier. Heat treatment of biomass is often recommended to increase the grindability of such fibrous materials. The present work is focused on the grindability of torrefied wood. To be representative of the grinding conditions, an impact device was designed and developed to evaluate the mechanical behavior of wood at high compression rates. This machine has been used to test two wood species (pine and oak) in two material directions (radial and tangential). Our results allowed the loss of mechanical strength caused by the torrefaction to be quantified. As intensity of the heat treatment increases, the material becomes more fragile and finally loses its fibrous behaviour which increases the number of peak events on the stress/strain curve and significantly reduces the deformation energy.

2.2. INTRODUCTION

In order to face to the fossil fuel crisis and the global warming, the use of renewable energy is promoted worldwide. Energy from biomass is one of this renewable energy, which has to be promoted as long as its production is not in competition with food production. Once dried, this energy is easy to store, which is a huge advantage compared to other renewable energy sources. Several sources of biomass, such as wood, straw, agriculture residues, wastes fulfill this criterion. This study is focused on woody biomass, which represents ca. 80% of the production of ligno-cellulosic materials in the world.

One interesting way to produce biofuel from biomass is gasification followed by a Fisher-Tropsch synthesis. However, fine particles are required to improve reaction rates, gas yield and flowability of materials inside the process (Simmons 1986; Bergman et al. 2005; Wei et al. 2006). As the fibrous and heterogeneous structure of biomass renders the grinding operation complex and demanding in energy, a pretreatment is required in this multistep process from biomass to liquid (BtL). In this domain, torrefaction seems to be a good way to facilitate this grinding step and to homogenize the technological properties of biomass.

Torrefaction consists in the exposition of biomass at temperature levels ranging from 180°C to 280°C in an inert atmosphere such as nitrogen (Bourgeois 1989). These conditions induce several chemical modifications of the cell wall, such as degradation of hemicelluloses (Avat 1993) and condensation of lignin (Weiland and Guyonnet 2003). Consequently, the technological properties are strongly affected: change in colour (Bekta and Niemz 2003), increase of durability against fungi (Hakkou et al. 2006), decrease of hygroscopicity and shrinkage (Almeida et al. 2009)…

Torrefaction also affects the mechanical properties of wood (Unsal and Ayrilmis 2005 ; Esteves et al. 2007) easing its grinding and reducing the need for grinding energy (Bergman et al. 2005; Repellin et al. 2010). For this purpose, the main technique used is to measure the power consumption of a grinder to grind heat-treated wood into a new product with well-known characteristics (particles size distribution, quantity of particles…). Nevertheless, rigorous and reproducible tests are still to be developed to quantify the effect of heat-treatment regarding grindability, namely as a function of species, temperature level and treatment duration; especially at a sample scale.

The aim of this paper is to assess the role of torrefaction on the grinding behaviour. In particular, to be closer to the grinding conditions, an impact device was specially conceived and developed to evaluate the mechanical behavior of wood at high compression rates (Pierre et al., 2011). Using this device, the grinding behavior of maritime pine (*Pinus pinaster*) and pedunculate oak (*Quercus robur*) torrefied under different extent of heat-treatment was determined in radial and tangential directions.

2.3. MATERIALS AND METHODS

2.3.1. Material selection and torrefaction procedure

The wood samples tested in this study were obtained from a maritime pine tree (*Pinus pinaster*), nearly 20 years old, from south-eastern France and a pedunculate oak tree (*Quercus robur*), nearly 35 years, from eastern France (Brin Forest).

Seven defect-free blocks per species with a straight grain angle and an annual growth width of nearly 3 mm were cut in the longitudinal direction of the log. The samples were stored under laboratory conditions before being oven-dried at 103°C (± 2 °C) up to equilibrium.

Then, the anhydrous mass (M_0) was determined to the nearest 0.001 g using a digital balance and the samples were stored in a desiccator containing silica gel until torrefaction. Torrefaction was performed with a device specifically developed in our laboratory for this purpose (Colin et al. 2007). Samples were treated at 220, 250 or 280°C for 1 or 5 hours under nitrogen according to the following protocol: (a) heating rate of 5 °C/min up to 100 °C; (b) plateau at 100 °C for 30 minutes; (c) heating rate of 5 °C/min until the final treatment temperature is reached then maintained at this level for 1 or 5 hours; and (d) decrease in temperature by 2 °C/min down to 150 °C (Pierre et al. 2011a).

After treatment, the anhydrous mass (M_{0T}) was measured in order to determine the mass loss caused by the heat treatment according to the following equation.

$$ML = \frac{M_{0U} - M_{0T}}{M_{0U}} \times 100 \tag{1}$$

where ML is the mass loss (%), M_{0U} is the initial oven-dried mass of the wood sample before heat treatment, and M_{0T} is the oven dried mass of the wood sample after heat treatment.

After torrefaction, 6 paired cubic samples with dimensions 10 × 10 × 10 mm^3 (R, T, L) were carefully sliced from these blocks using a diamond wire saw. All samples had the same number of latewood layers with roughly the same ring width.

The upper surface of each sample was prepared for observation using a slide microtome (*Microm HM 440E*). The average basic wood density (oven-dry mass to green volume) of heat treated wood samples was determined according to standard methods.

2.3.2. Mechanical properties

The grindability of torrefied wood has been assessed by the determination of its mechanical behavior under high compression rates. A new experimental impact device has been used for this purpose. (Pierre et al., 2011b). This impact system consists of a moving trolley, equipped with an accelerometer, which is thrown against a fixed trolley. The sample is attached to the fixed trolley and the acceleration of the moving trolley during the impact is integrated twice to obtain the strain/stress curve. A high speed camera synchronized with a high-powered xenon flash records up to 4000 frames/second (figure IV-1). More detail about the technical aspects of this device may be found in Pierre et al. (2011b).

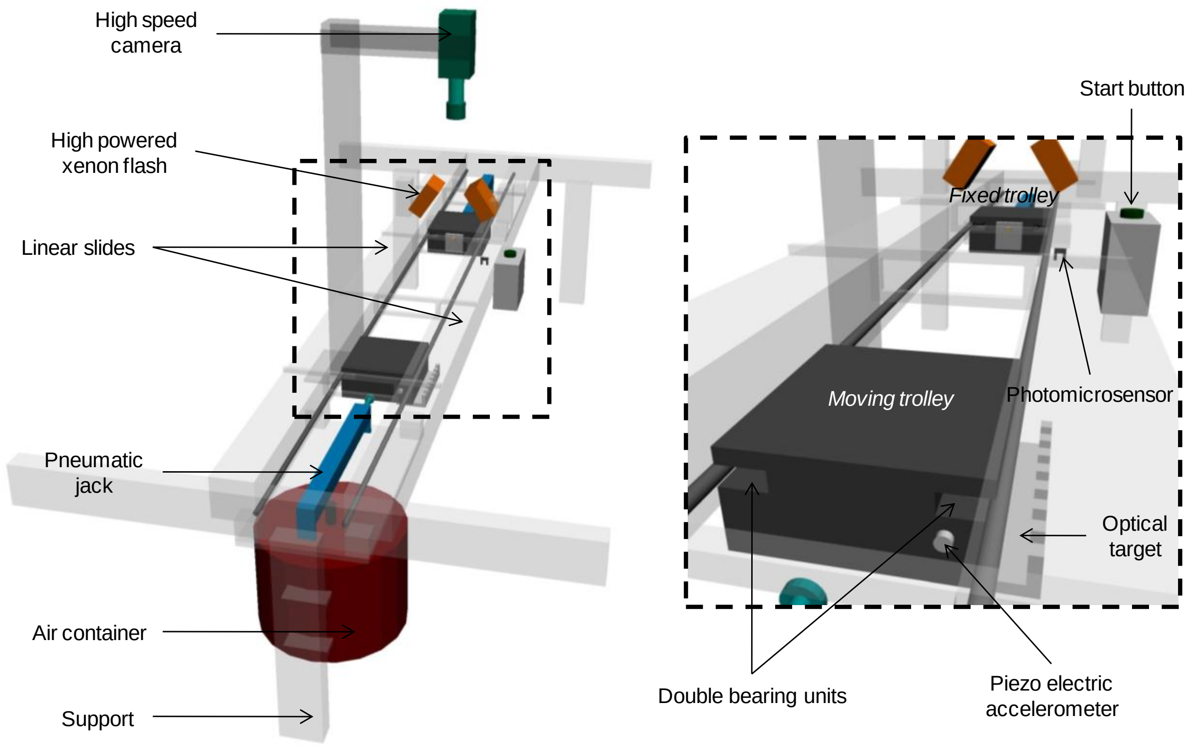


***Figure IV-1.** Experimental set-up. A) General view; B) Zoom of the trolley and sample fixation (Pierre et al,.2011b).*

Samples of heat treated maritime pine and pedunculate oak have been tested under radial and tangential directions. The impact velocity was ca. 1.7m/s.

The calibrated instrumentation, namely accelerometers and contactless position sensors, allow the stress/strain curve to be obtained with a good accuracy. Theses curves are then analyzed to extract some mechanical properties. Because of the oscillations of the stress/strain curves, it was difficult to determine the Young's modulus especially for samples treated with the highest treatment extend (highest values of duration and temperature). Thus, only the average stress level during the compression plateau, the absorbed energy and the brittleness were determined.

The stress level of the compression plateau was determined as the average stress level between 25% and 35% strain. The peaks on the stress/strain curve are used as indicators of the brittleness of the sample. A specific way of dealing with them has been devised, the idea being to report both peak number and intensity. The peaks are first located and counted then their amplitude is determined. We developed an algorithm doing this by searching for all the points on the stress/strain curve. A new peak is defined as soon as the stress level of the next point is smaller than the current point. From this current point, the algorithm continues to test successive points while the stress level continues to decrease. Once the lowest level has been attained, the amplitude of that peak A_P is defined as the difference in the stress value at the current point minus the smallest value, attained just before the stress increases again. When all the peak magnitudes have been detected, they are sorted over the values ranging from val_{min} to val_{max} in a number N of categories, either on a linear or a logarithmic scale.

val_{min} is not necessarily the smallest peak value encountered in a given test. On the contrary, we can choose a threshold value slightly higher than the noise level of the device. This noise value is assessed from the amplitude of the peaks obtained on the signal during free displacement of the moving trolley (Pierre et al., 2011b). Results of the peak analysis are given through classical curves. Each point represents the average of number of peaks, in a same range of amplitude.

The absorbed energy per unit volume E is obtained by the define integral of the stress over the strain:

$$E(\varepsilon) = \int_0^{\varepsilon} \sigma(\varepsilon) d\varepsilon \qquad (2)$$

With: E the absorbed energy (MJ/m^3), σ the stress (MPa) and ε the deformation.

From the experimental data, a discrete integration was used to compute this function:

$$E(\varepsilon_{n+1}) = \sum_{i=0}^{n} \frac{\sigma_{i+1} - \sigma_i}{2} \times \left(\varepsilon_{i+1} - \varepsilon_i\right) \qquad (3)$$

2.4. RESULTS AND DISCUSSION

Table IV-1 depicts the mass loss of heat-treated maritime pine and pedunculate oak. As expected, the mass loss increases with duration and temperature of treatment in both species. This mass loss is due to the production of volatiles during the chemical degradation of wood components (Bourgois and Guyonnet 1988; Avat 1993; Sivonen 2002; Pierre et al. 2011). Due to its ability to consider the cumulated effect of both temperature level and treatment duration into consideration, the mass loss is a good indicator of the treatment severity (Almeida et al. 2010; Pierre et al. 2011).

A significant difference exists between the two species: the mass loss for oak being always higher than for pine for a given treatment level. For example, the more severe treatment (280°C; 5h) caused a mass loss of 38% for maritime pine compared to 45.2% for oak wood. Indeed, hardwood species are well-known to be more sensitive to temperature than softwood species, regarding either thermal activation (Olsson and Salmèn, 1992, Placet et al. 2007) or thermal degradation (Avat, 1992, Esteves, 2007). For example, Pierre et al. (2011) confirmed that the higher mass loss of oak is due to the easier chemical degradation of hardwood components, namely hemicelluloses.

Table IV-1. *Mass loss of heat-treated maritime pine and pedunculate oak.*

	Temperature (°C)	Time (h)	Mass loss (%)	Average density (kg/m^3)
Maritime pine	Untreated		-	441.7
	220	1	2.7	390.2
	220	5	5.4	406.2
	250	1	9.3	397.8
	250	5	16.1	392.0
	280	1	23.2	381.0
	280	5	38.0	324.3
Pedunculate oak	Untreated		-	624.0
	220	1	6.4	625.2
	220	5	12.8	644.1
	250	1	17.8	640.8
	250	5	23.0	605.3
	280	1	31.1	588.0
	280	5	45.2	543.1

As the mass loss is only partly compensated by the reduction of sample size, the average density globally decreases with increasing duration and/or temperature.

The images of the sample surface grabbed during the test thanks to the high speed camera are highly informative and are worth an initial qualitative analysis. Figure IV-2 to figure IV-5 present a selection of images taken during the impact tests.

Globally, the effect of the treatment intensity is clearly depicted. Whatever the direction and the species, the increase of treatment intensity increases the brittleness. Contrary to untreated samples which display a high resilience, samples treated at the highest extents of treatment (280°C; 5h) are crushed into small particles forming a cloud around the rest of the sample. Samples treated at an intermediate treatment (250°C; 5h) depict an intermediate behavior which produces larger particles. A simple observation of the samples after the test confirms these preliminary observations (figure IV-6). The proportion of small particles increases with the treatment intensity. Note that oak seems to be more affected than pine as the increase of the number of small particles seems to be higher for this species. This is obviously a direct effect of the best resistance of softwood macromolecules to high temperature, as explained above. However, this result can also be partly explained by the differences in anatomical structure. Tracheids in softwood (2 to 4 mm) are indeed ca. three times longer than libriform fibers of hardwoods. In addition, the presence of wide vessels in oak forms zones of low mechanical resistance which allow cracks to be easily initiated. These explanations are in agreement with the results previously obtained by Repelin et al. (2010)

who showed that the average particle size from beech and spruce decrease with increased extent of treatment. According to the general context of this study, the increased proportion of small particles is a positive result as it guarantees a good flowability of the powder and a rapid reaction rate. Bergman et al. (2005) showed that the optimal particle size for this purpose is about 100 µm. The images of untreated samples are not presented in figure IV-6, since these samples remained undivided after the test.

Figure IV-2 to figure IV-5 also give some information about fractures mechanisms occurring within the sample. For untreated samples and samples with a moderate treatment, the deformation is the result of the accumulation of local deformation at zones distributed throughout the specimen. In the case of radial compression, the earlywood layers of maritime pine deform first, due to their low density. The large compression of these zones produces an impressive Poisson's effect that can be easily observed on the sample edges, at the earlywood/latewood transition. This explains why radial fractures appears somewhere between earlywood and latewood, where the heterogeneity of Poisson's effect is the highest. For pedunculate oak, the fractures appear along the large thick rays and are propagated along the untreated sample and the moderately treated sample. Large particles are formed showing a loss of resilience.

In the case of tangential compression, the weak zones of the sample are always the earlywood layers, either for pine or for oak. Therefore, in this direction, the weak and strong layers act in parallel. This absence of cohesion in the direction parallel to the deformation explains why some large particles are ejected from the edges when the compression wave passes through the sample (untreated, image 4 of figure IV-3 and 250°C/5 h of figure IV-5). In general, the cohesion seems to be weaker for pine: even for untreated samples, the compression at the fixed plate widens the sample which takes a trapezoid shape. Once initiated, this opening progresses and opens tangential fractures through the weak layers, which separate the annual rings. This mechanism is observed whatever the treatment intensity for pine: it just arrives earlier as the treatment intensity increases. In the case of oak, this mechanism clearly appears only for the most severe treatment (280°C/5 h, image 3 of figure IV-5). For lower treatments (untreated and middle treatment), oak tends to keep its cohesion and the sample shape remains parallelipedic throughout the test. This is probably due to the presence of large rays that reinforce the ring-porous zone in radial direction. The sample treated at 250°C/5 h starts to be divided in large particles at 1.25 ms, just followed by parallel checks straight through the sample. These checks follow the large rays in the radial direction and some tangential checks appear also at the end of the test in the ring-porous zones.

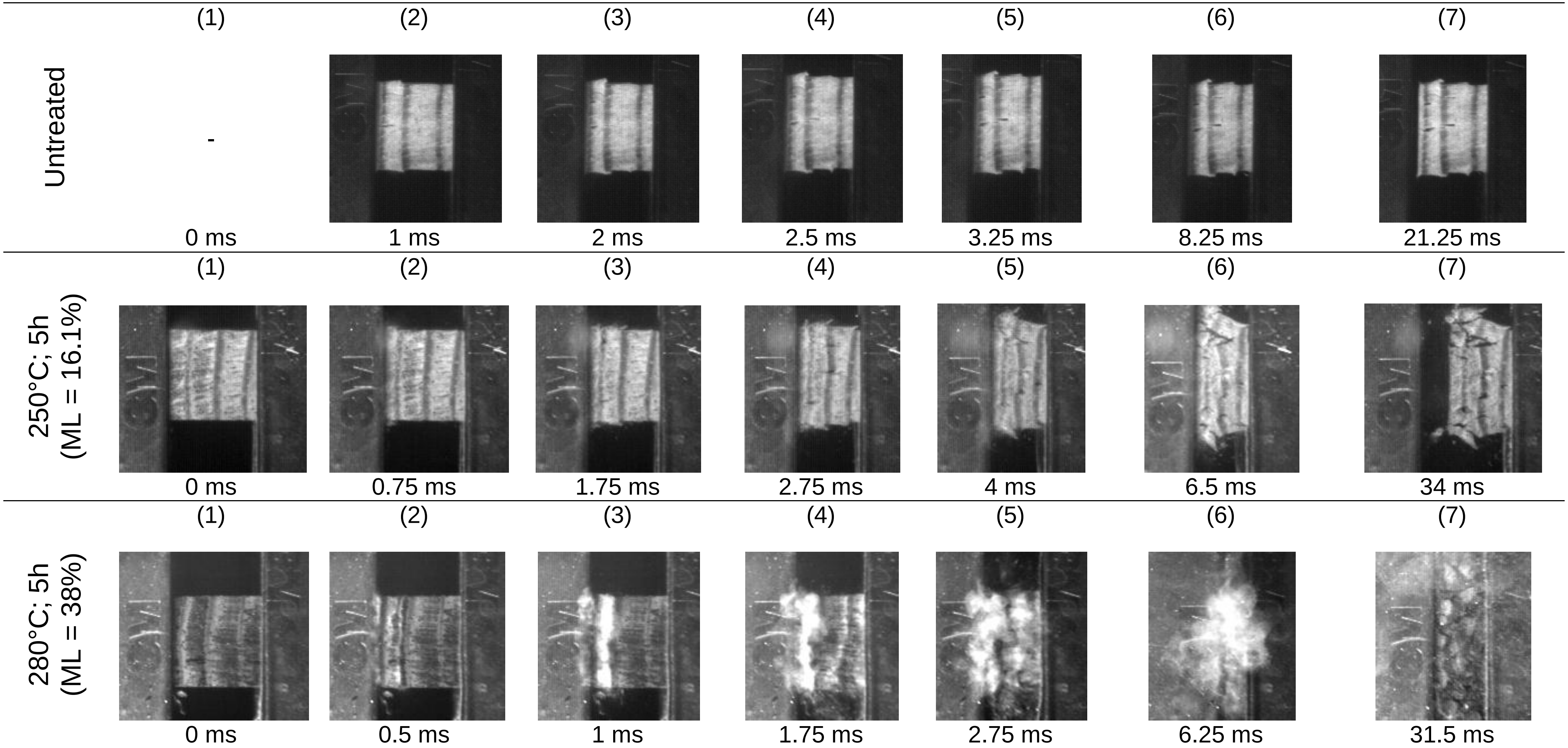


Figure IV-2 *Deformation of untreated and heat treated maritime pine during dynamic compression (radial direction).*

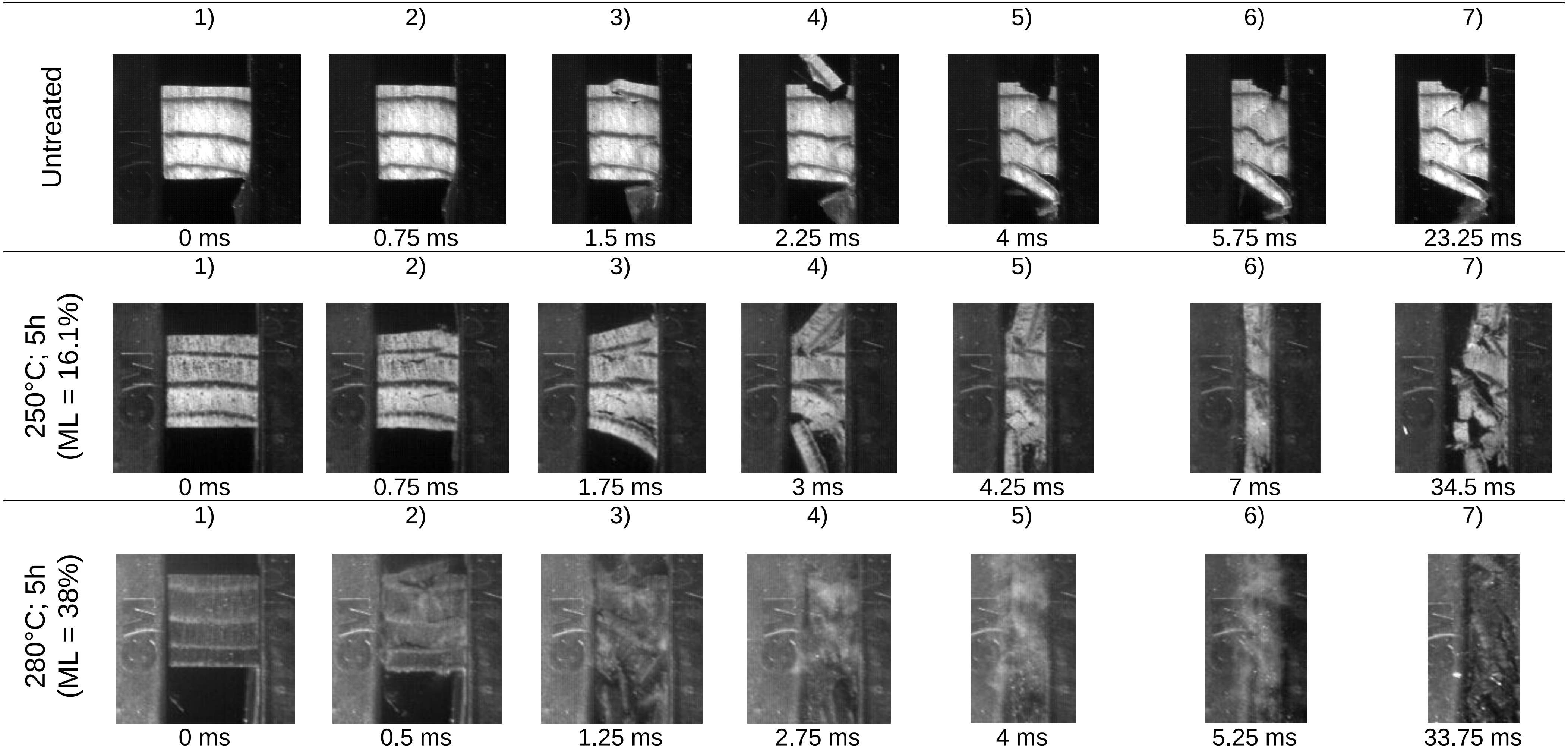


Figure IV-3. *Deformation of untreated and heat treated maritime pine during dynamic compression (tangential direction).*

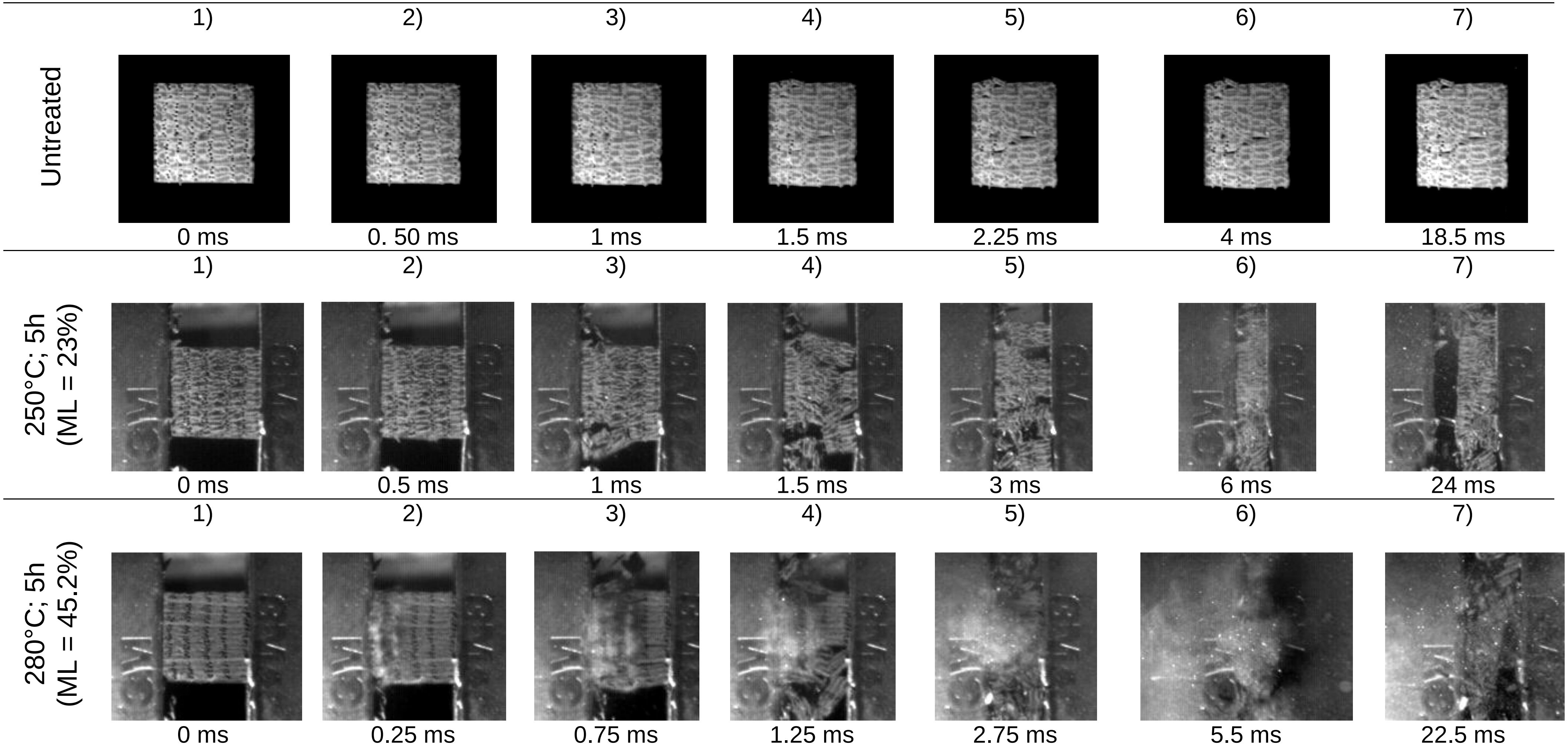


***Figure IV-4**. Deformation of untreated and heat treated pedunculate oak during dynamic compression (radial direction).*

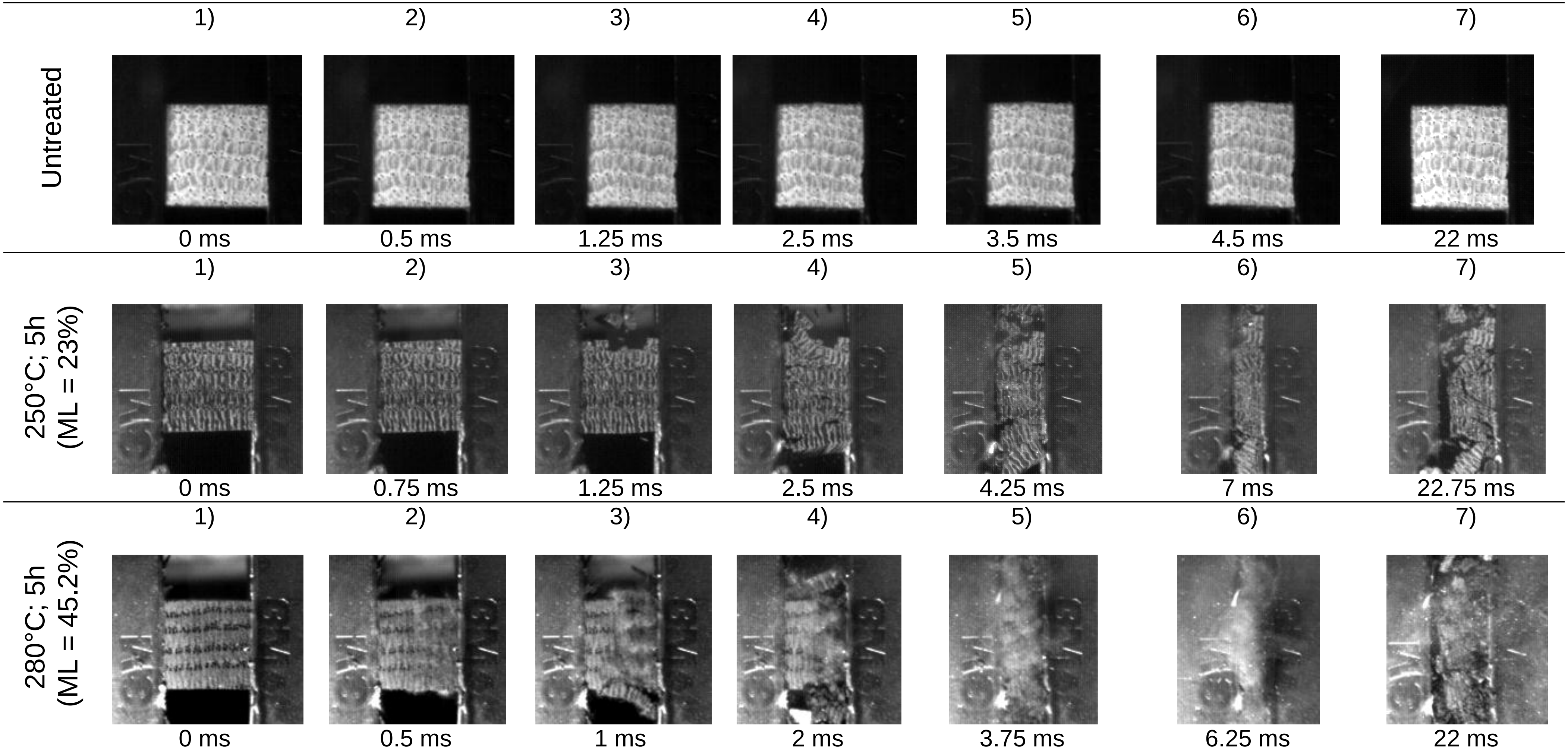


Figure IV-5. *Deformation of untreated and heat treated pedunculate oak during dynamic compression (tangential direction).*

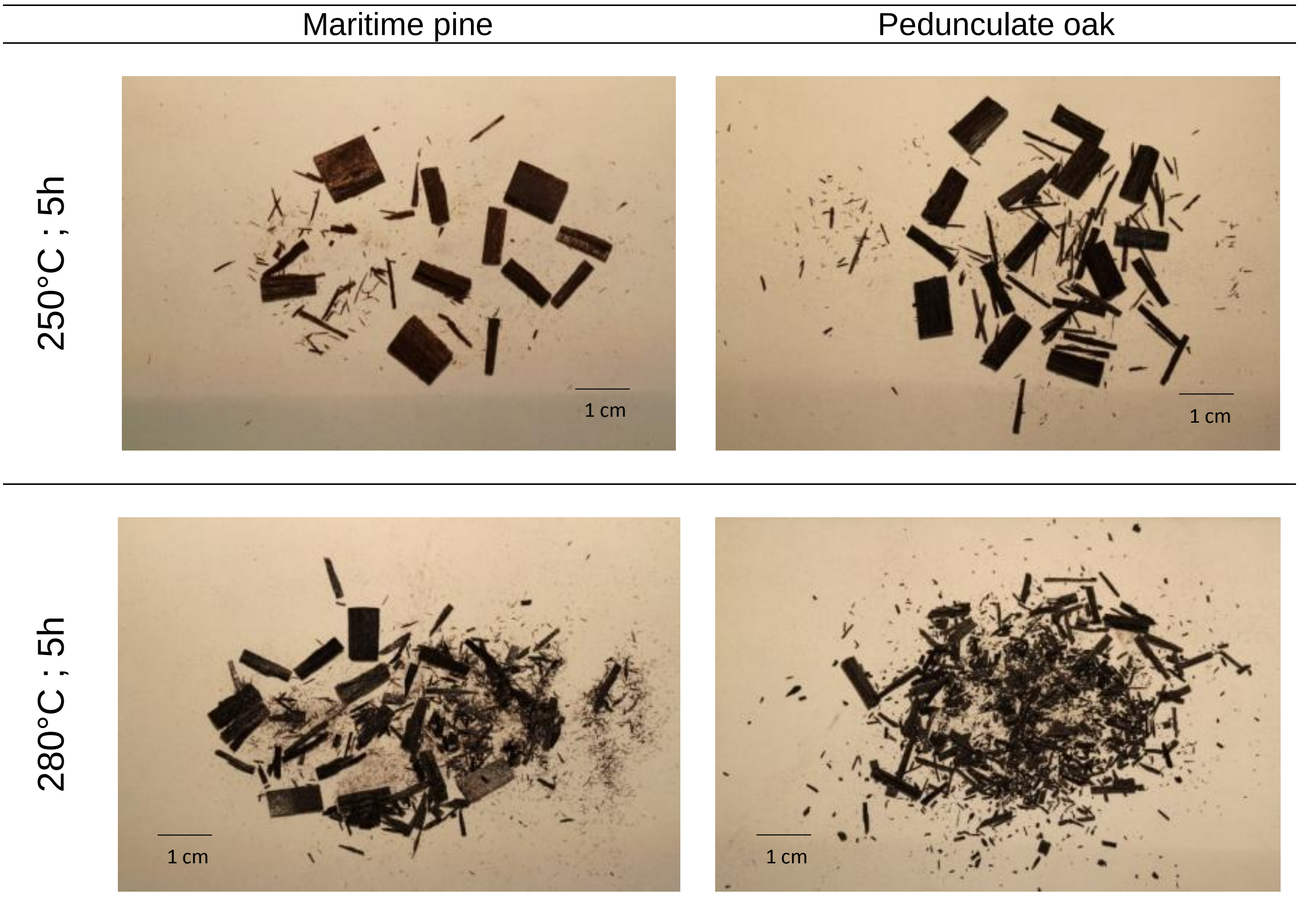


***Figure IV-6.** Heat treated wood particles after impact (tangential direction).*

Figure IV-7 depicts the fracture facies of particles collected after the impact. Two thresholds can be distinguished in the grinding behavior of heat treated biomass. For native samples and low heat treatment, the product keeps a resilient behavior, able to keep the sample integrity in the present study and producing curved rupture facies in particles obtained by grinding (Almeida et al. 2009). After this threshold, the product behavior turns towards an elastic-fragile behavior, with clean rupture facies such as those that can be observed at 250°C/5 h. In this domain, the particles keep an elongated shape, as the energy required to split the fibrous structure remains smaller than the energy required to break the fibers. When the treatment severity continues to increase, a second threshold may be reached, where the fibrous behavior of the lignocellulosic material vanishes. This stage is attained when the brittleness is such that the breaking energy approaches the splitting energy. The most obvious effect of this regime is the production of particles with a small elongation ratio. This domain is clearly obtained for oak at 280°C/5 h, which produces very short particles in the longitudinal direction (figure IV-7). This regime is looked for to supply a gasifier because spherical particles ease the powder. Obviously, in order to optimize the raw material, it is best if this regime is obtained with the minimum mass and/or energy loss.

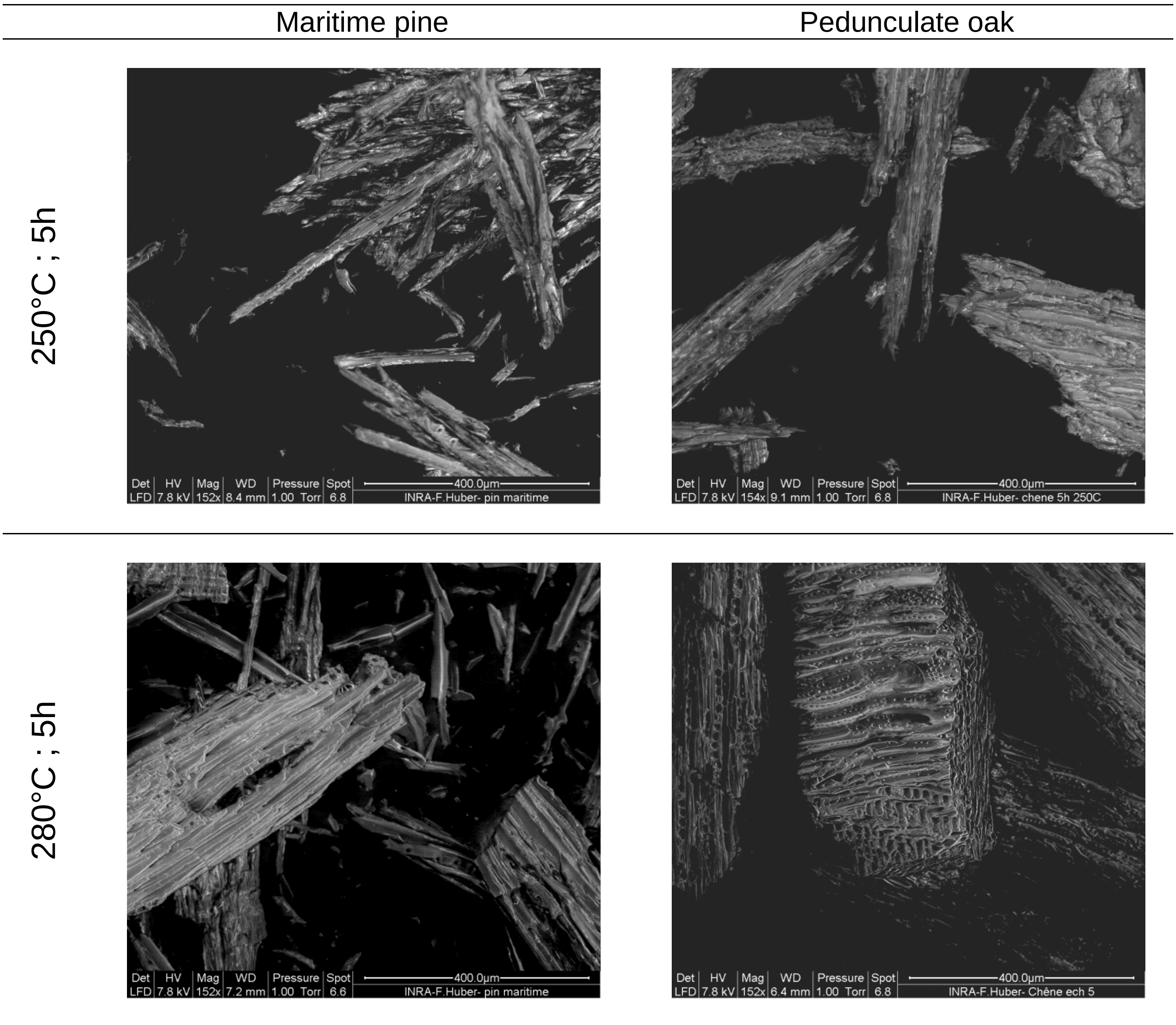


Figure IV-7. *Fracture faciès of heat-treated wood submitted to impact device.*

Figure IV-8 presents a set of raw stress/strain curves obtained for maritime pine. This figure confirms that the untreated pine sample exhibits a resilient behavior under dynamic radial compression whereas heat treated pine samples exhibit a brittle behavior. Notice that, due to the principle of our device, the maximum deformation energy is determined by the initial kinetic energy stored in the moving mass: therefore, the maximum deformation increases at decreasing stiffness. This explains why the maximal deformation increases from ca. 30% to 80% with the treatment severity. For the same range of heat treatments, the stiffness, determined by the first linear part of the stress/strain curve decreases from 675 to 57 MPa. The behavior in tangential direction follows the same trends, even though the tangential checks along the earlywood layers hide some evolutions.

The stress level of the compression plateau also decreases at increasing treatment intensity (table IV-2). The compression plateau corresponds to an inelastic domain which results from collapse and gross deformation of the cell (Easterling et al. 1982). The chemical degradation of wood components induces general degradation of cell walls easing the breakability of cells. This is especially marked for the more severe treatment, where the compression plateau exhibits a dramatic reduction. The compression plateau contains several peaks which is an indication of the brittleness. At first glance, figure IV-8 already exhibits a clear influence of the treatment extent. It can be noticed that few peaks of large amplitude are present for the untreated sample (below 20% of deformation) whereas treated samples depicts a large amount of peaks with low amplitude, over a wide range of deformation, especially from 20%.

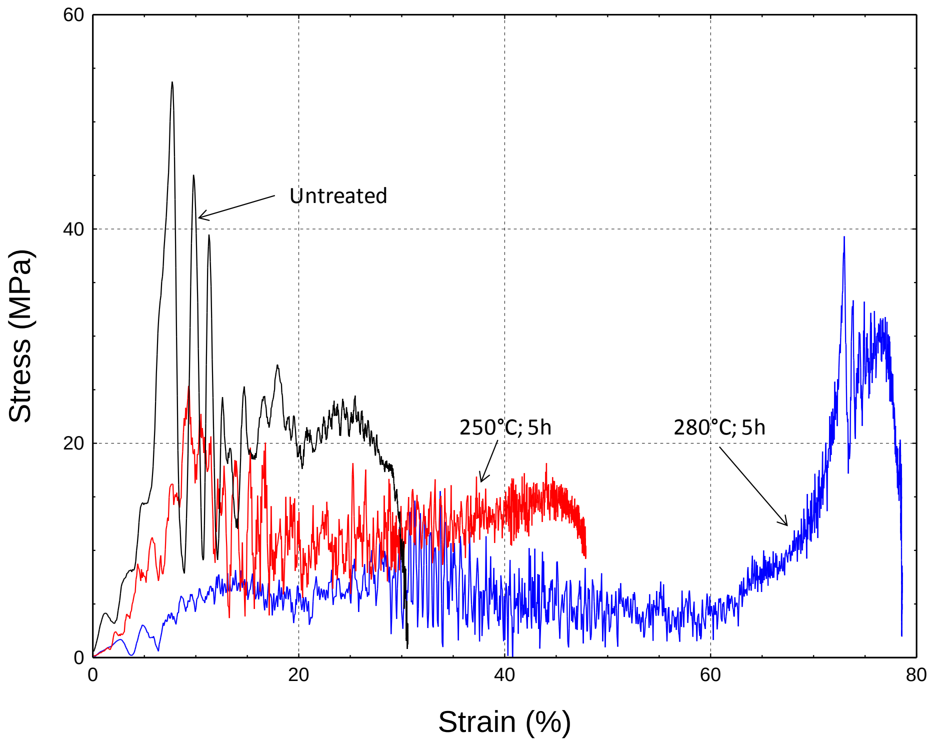


***Figure IV-8.** Deformation of heat treated maritime pine during dynamic compression along radial direction.*

In order to go further in the interpretation of curves obtained by the impact tests, the peak analysis is very informative (figure IV-9 and figure IV-10). In these figure, the peak profile is indeed the average of three repetitions, for each treatment extend. An amplitude range from 1.2 to 82 MPa and a logarithmic scale ($0.18 < \ln(A_P) < 4.41$) was selected, which allows:

- the largest peak amplitudes observed on our results to be included,
- the small peaks, observed on blank tests, hence related to the intrinsic noise of the apparatus, to be removed,
- the large peaks to be merged together thanks to the logarithmic scale.

However, it is still very difficult to distinguish the very few peaks of large amplitude, compared to the large number of small peaks. The discussion will therefore be more focused on the peak intensity profile in the range 1.2 to 22.4 MPa ($0.18 < \ln (A_P) < 3.11$).

At first glance, no monotonic trend can be observed from native wood to sample with a severe heat treatment, as the 280°C/5h peak profile seems to come back close the profile obtained for the native sample. A consistent change of the behavior can nevertheless be clearly stated.

Native wood has a huge resilience : the kinetic energy stored in the moving mass can therefore barely compress the sample. The resilience allows a large deformation energy to be stored within the sample and to be suddenly released a few times, producing some peaks (1 to 3) with large amplitudes. These releases are certainly initiated by the formation of radial checks, in the earlywood/latewood transition zone in the case of pine and in the large rays in the case of oak. No peaks of intermediate intensity are observed and a certain number of peaks with low amplitude, occurring at the end of the test, raise the peak profile on the left side. The presence of small peaks at the end of the test is certainly a cumulative effects of small releases in the sample (occurring at the level of cell layers) and of device vibrations at the end of the test.

At increasing treatment intensity, the sample become more brittle and the amount of energy stored between two successive releases decreases. As the kinetic energy is the same for all tests, the number of peaks necessarily increases. This explains why the intensity profiles present a maximum, whose value increases and whose position shifts towards lower amplitude values as the treatment intensity increases. For the most severe treatment (280°C/5 h), this maximum finally disappears as it merges with the left rise always present, but with a larger probability at increasing treatment extents. Note that this general trend is most obvious for tests in radial direction, where weak and strong layers act in series, which promotes local failure. Indeed, even brittle samples depict a global failure for tangential test (weak layers along the test direction), which is not suitable for a good discrimination of the brittleness (figure IV-3 and figure IV-5).

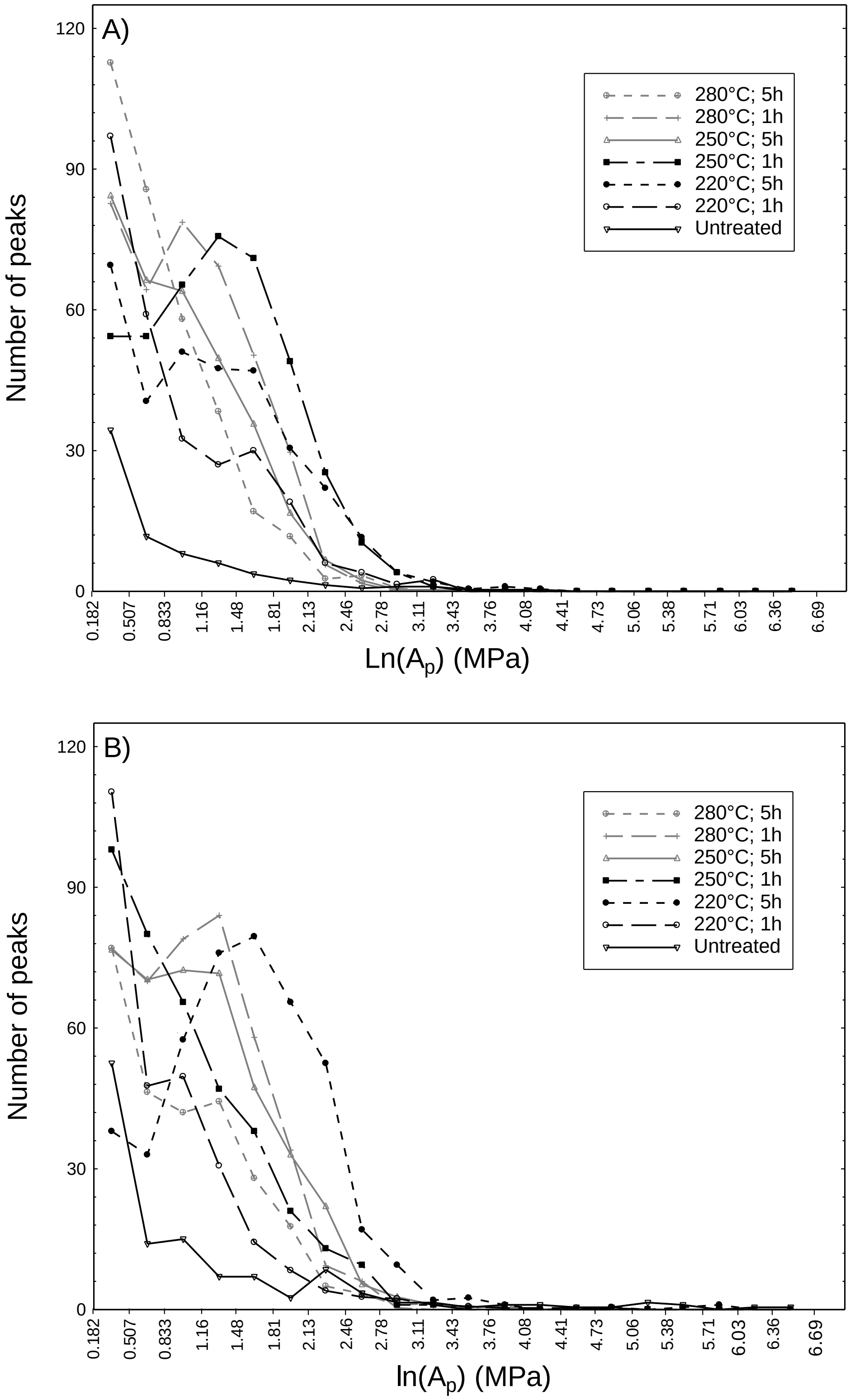


Figure IV-9. *Grindability of heat treated maritime pine A) Radial compression; B) Tangential compression.*

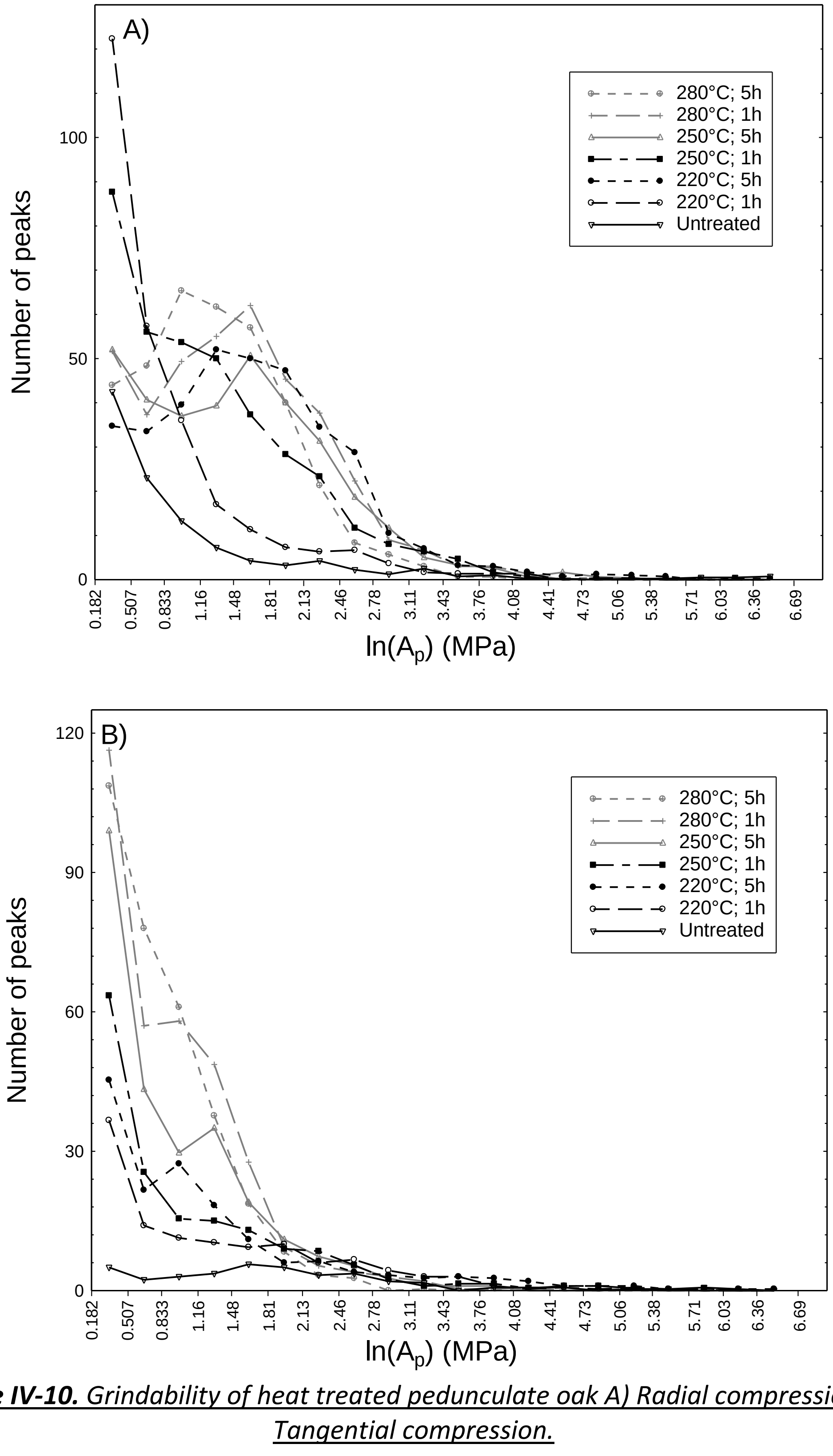


***Figure IV-10.** Grindability of heat treated pedunculate oak A) Radial compression; B) Tangential compression.*

Figure IV-11 and figure IV-12 show the absorbed energy (E), either stored or lost, as a function of deformation. A general stratification appears, with curves with a lower average slope at increasing treatment extend. Because almost the same impact velocity was used for all tests, it is consistent to notice that the final absorbed energy is very similar (about 6

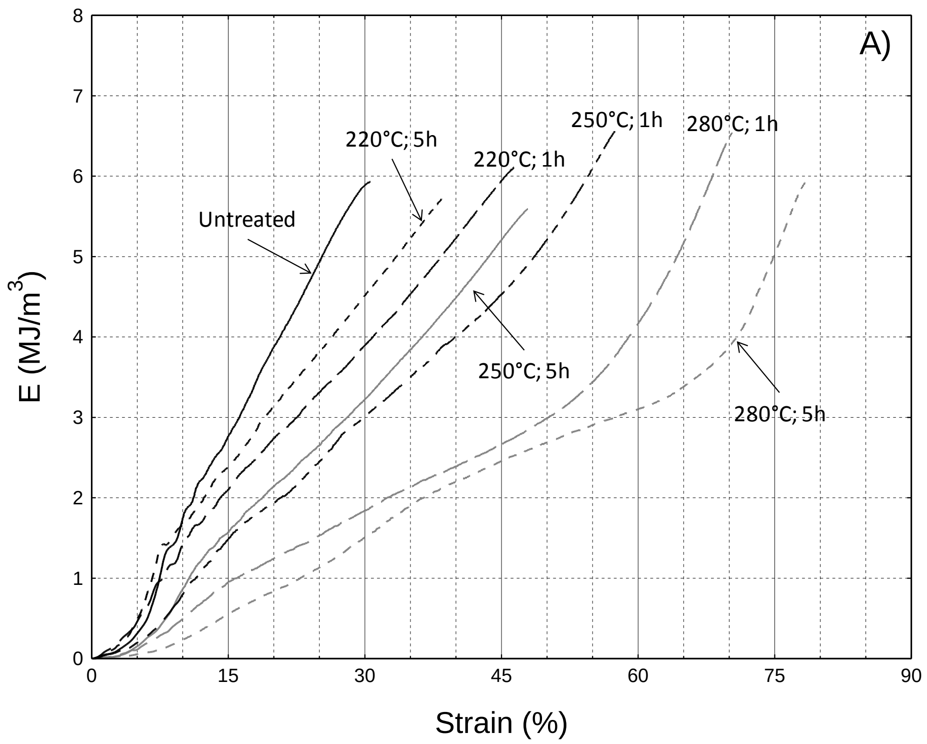
MJ/m^3). A direct relationship exists between the brittleness and the maximum deformation at the end of the test.

All curves depict the same global shape. At low deformation values, the curves present a parabolic shape, consistent with an elastic behavior, where the stress increases linearly with the strain level. This initial shape almost disappears for the samples treated at 280°C.

Whatever the extent of treatment, the absorbed energy at 5% of deformation ($W_{5\%}$) decreased at increasing treatment extent whatever the species and the direction (table IV-2). In the case of pedunculate oak, the large rays contribute to the wood stiffness in radial direction and then to the energy absorption during the elastic domain. Until the highest extent of treatment, the absorption of energy is higher for pedunculate oak than for maritime pine in radial direction. In tangential direction, the rays have no influence on the energy absorption. Thus no clear difference is depicted between maritime pine and pedunculate oak in this direction.

Then elastic zone is followed by the compression plateau where the increase of energy is roughly linear. For samples treated at the lowest extent of treatment, this absorption is due to fracture dissipation and to collapse deformation of cells whereas it is more due to fracture deformation and to a spatial rearrangement of particles formed for samples treated at the highest extents.

Finally, its is important to notice that the samples treated at 220°C and 250°C absorb more energy for a 5-hour treatment than for a 1-hour treatment. This surprising behavior could be explained by a reorganization of macromolecules after a certain time, namely lignin condensation, which increases why the apparent stiffness increases with the treatment duration after the initial decreases (Assor et al. 2009, Rousset at al. 2009). Note that this additional absorbed energy will eventually turns into a larger production of fine particles, which could justify a long treatment.



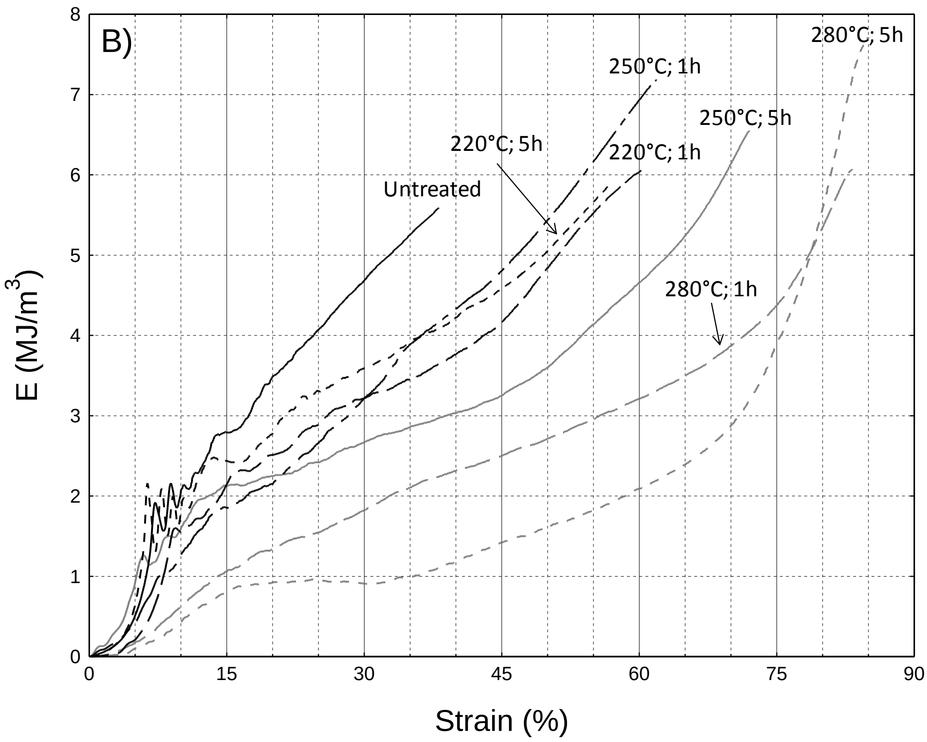


Figure IV-11. *Absorbed energy versus deformation for maritime pine (A: radial compression; B: tangential compression).*

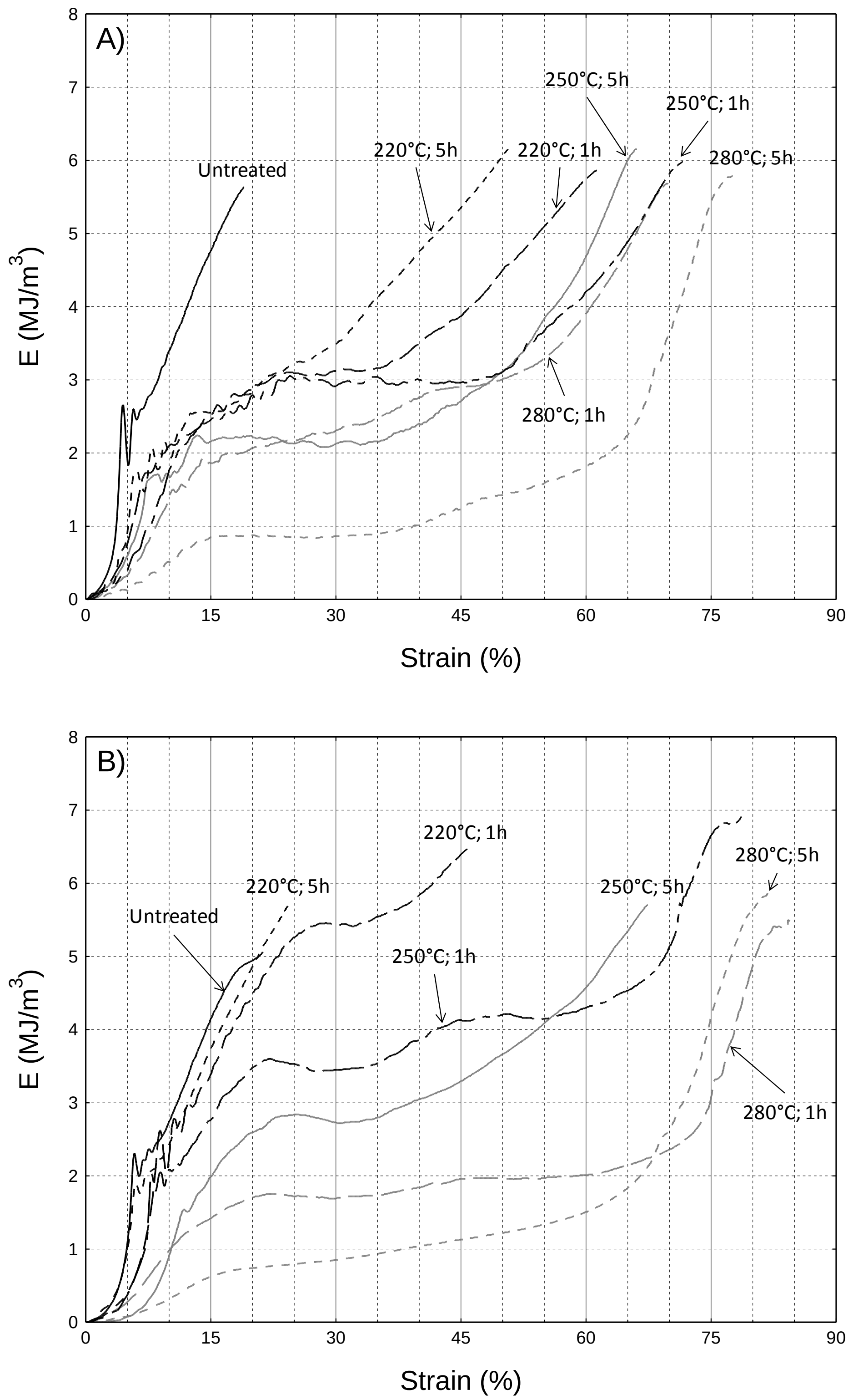


***Figure IV-12.** Absorbed energy versus deformation for pedunculate oak (A: radial compression; B: tangential compression).*

***Table IV-2**. Some mechanical properties of heat treated maritime pine and pedunculate oak tested along radial and tangential direction.*

			Radial compression			Tangential compression		
	Temperature (°C)	Time (h)	$E_{5\%}$ (MJ/m^3)	$E_{25\%}$ (MJ/m^3)	σ_{pl} (MPa)	$E_{5\%}$ (MJ/m^3)	$E_{25\%}$ (MJ/m^3)	σ_{pl} (MPa)
Maritime pine	Untreated		0.3 (0.1)	4.5 (0.6)	20.3 (0.3)	0.5 (0.1)	4.1 (0.0)	15.1 (4.4)
	220	1	0.3 (0.2)	3.2 (0.2)	11.7 (0.1)	0.5 (0.4)	2.7 (0.3)	5.6 (1.3)
	220	5	0.4 (0.1)	3.8 (0.1)	13.6 (0.3)	0.6 (0.0)	3.0 (0.5)	7.5 (0.9)
	250	1	0.2 (0.1)	2.4 (0.2)	10.1 (1.0)	0.5 (0.1)	2.7 (0.1)	7.9 (3.9)
	250	5	0.2 (0.0)	2.8 (0.3)	12.2 (1.3)	0.5 (0.4)	2.3 (0.6)	3.5 (1.9)
	280	1	0.1 (0.0)	1.6 (0.0)	5.8 (0.2)	0.2 (0.0)	1.5 (0.1)	4.9 (0.9)
	280	5	0.1 (0.0)	1.2 (0.1)	6.3 (0.8)	0.1 (0.0)	0.8 (0.1)	0.6 (0.6)
Pedunculate oak	Untreated		2.1 (0.4)	-	-	0.8 (0.3)	-	-
	220	1	0.7 (0.1)	3.2 (0.7)	4.3 (1.7)	0.5 (0.1)	4.3 (0.9)	5.3 (4.7)
	220	5	1.5 (0.4)	3.8 (0.9)	6.1 (3.5)	1.1 (0.4)	-	-
	250	1	0.4 (0.1)	2.8 (0.7)	4.0 (4.6)	0.4 (0.0)	3.8 (0.4)	5.1 (7.6)
	250	5	0.5 (0.2)	2.5 (0.6)	4.3 (6.9)	0.3 (0.2)	3.0 (0.3)	3.0 (2.4)
	280	1	0.2 (0.1)	2.0 (0.3)	1.1 (1.5)	0.3 (0.1)	1.6 (0.2)	-0.5 (0.5)
	280	5	0.1 (0.5)	0.8 (0.1)	0.3 (0.4)	0.1 (0.0)	0.7 (0.1)	0.6 (0.5)

Values in brackets represent the standard deviation based on duplicated tests.

2.5. Conclusion

The grindability of heat treated maritime pine and pedunculate oak has been assessed with the aid of an original impact device conceived and developed in our lab. Our results showed that the grindability increases at increasing extent of treatment for both species. Global observations of the behavior of heat treated sample during an impact test were confirmed by mechanical analyses.

The treatment extend always increase the brittleness of wood. Starting from the native, air-dried state, the heat treatment induces a stepwise change of the mechanical behavior: the sample losses its resilience, then its fibrous behavior. The second step is required when the goal of heat treatment is to increase the grindability of wood and to obtain rather particle with a small elongation ratio.

Contrary to many other properties, whose alterations by heat treatment is nicely predicted by the global mass loss, the results presented in this work seems to prove that two parameters (temperature level and treatment duration) should be distinguished to control the change of mechanical behavior.

2.6. ACKNOWLEDGMENT

This work was financially supported by the ANR project TORBIGAP. The authors are grateful to the personnel of the LERFoB 3B team.