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Response of mechanical and thermal properties of a vibration-welded polypropylene/SiO₂ nanocomposite on different thermal treatments

Effects of post-processing thermal treatments of vibration-welded polypropylene and its silica nanocomposite were systematically studied by using a heating cabinet and in-line infrared radiation. Experimental results demonstrate that thermal treatments, independent on the heating sources, exert obviously positive effect on improving the weld quality of polypropylene materials, i.e. stiffness and strength of the weld. Despite the short cycles used for infrared heating, the results are better and the effort is less.

Einfluss verschiedener Wärmebehandlungen auf die mechanischen und thermischen Eigenschaften eines vibrationsgeschweißten Polypropylen/SiO₂-Nanokomposits

Der vorliegende Beitrag untersucht systematisch den Einfluss einer thermischen Nachbehandlung mit Hilfe eines Wärmeschranks sowie einer Inline-Infraroterwärmung auf die Eigenschaften eines vibrationsgeschweißten Polypropylens und eines PP/SiO₂-Nanokompositen. Die Ergebnisse zeigen, unabhängig von der Wärmequelle für die Temperung, eine Verbesserung von Elastizitätsmodul und Festigkeit der Verbindung. Trotz der kurzen für die Infraroterwärmung benutzen Zyklen sind hier die Resultate besser und der Aufwand geringer.

Response of mechanical and thermal properties of a vibration-welded polypropylene/SiO₂ nanocomposite on different thermal treatments

L. Y. Lin, A. K. Schlarb

1 INTRODUCTION

Vibration welding is a commonly used technique for joining thermoplastic materials for manufacturing hollow parts in the industrial production. Effects of the welding parameter on the final weld quality have been comprehensively investigated both for non-reinforced and reinforced polymeric materials [1-6]. According to Schlarb and Ehrenstein [1], the vibration welding process can be divided into four stages, which are i. solid friction, ii. non-steady stage of melt generation, iii. quasi-stationary melt generation and iv. holding phase. The stage iii is the crucial phase for determining the mechanical properties of the joint. The weld exhibits quite poor mechanical properties without reaching this stage. In addition, in order to achieve a weld factor of one, which means that the weld strength is equal to that of the bulk material, the lateral elongation rate of the melt flow within the weld region during the welding process must be lower than a critical value, which is dependent on the polymeric materials needed to be joined [7].

In the last decades, studies on vibration welding of a novel material class, i.e. thermoplastic-based nanocomposites with spherical nanoparticles or nanoparticles with low aspect ratio, showed that the weld strength was lower than that of non-reinforced one, which means that the reinforcing effect of such nanoparticles was not exploited into the weld. The reasons of this phenomenon can be attributed to the agglomeration of nano-sized particles [8] and orientation of needle-shaped nanoparticles [9] in the weld region as well as the narrow weld region induced by decreasing viscosity after incorporating nanopartilces [10]. In contrast, vibration welding of nanocomposite filled with carbon nanotubes, which have huge aspect ratio, presented exiting results. A welding factor of one was obtained. Bridging of the carbon nanotubes cross over the weld region is responsible for this excellent weld strength [11].

In this study, vibration welding of polypropylene (PP) and its nanocomposite filled with spherical silicon dioxide nanoparticles was performed. After the welding process, the weld was thermally treated by using a heating cabinet and in-line infrared radiation, in order to assess the effects of the annealing process on the weld quality.

2 EXPERIMENTAL

2.1 Materials and nanocomposite preparation

To study the effect of the thermal treatments on the mechanical and thermal properties of vibration-welded polypropylene nanocomposite, a commercially available isotactic propylene (iPP), HD120MO, Borealis, Austria, was chosen as polymer matrix. Hydrophobic silicon dioxide (SiO₂, Aerosil R8200, Evonik Industries, Germany), served as nano-size fillers in the PP matrix. According to the manufacturer, the SiO₂ content for this grade of nano-SiO₂ is $\geq 99.8\%$. The specific surface area of the nanoparticles is $160 \pm 25 \text{ m}^2/\text{g}$. Compounding of the nanocomposite was conducted on a co-rotated twin-screw extruder (TSK-N 030, Theysohn Extrusionstechnik GmbH, Germany) with screw speed of 160 rpm. The screw diameter is 30 mm and it has an L/D (length/diameter) ratio of 40. The temperature zones were set from 190 °C near the hopper to 210°C at the die [12]. Designations of the materials studied are summarized in Table 1. In addition, nanofiller content in this work was kept constantly as 1 vol.-%.

Designation	Polymer matrix	Nanofillers	Dimension of nanofillers	Filler content, vol.-%
PP	Polypropylene	-	-	-
PP-S	Polypropylene	Nano-SiO ₂	Spherical, 12 nm	1

Table 1: Designations of the polypropylene-based nanocomposites

After preparation of the materials, the granules prepared via twin-screw extruder were injection-molded to plates with a dimension of 50 mm x 50 mm x 4 mm for vibration welding by using an Engel victory 80 injection-molding machine, Engel Austria GmbH, Austria. Injection-molding parameters are summarized in Table 2.

Material	T _{mold} °C	T _{zone1} °C	T _{zone2} °C	T _{zone3} °C	T _{zone4}	T _{zone5} °C
PP/PP-S	40	170	180	190	190	190

Table 2: Injection-molding parameters

2.2 Vibration welding

Welding of the injection-molded plates were carried out on a Branson vibration-welding machine (M-112H, Branson Ultraschall, Germany) under optimized processing conditions, as ascribed in the section of introduction. The weld of pure PP prepared under these conditions exhibits a weld factor of one. In addition, PP-S weld presents also the best mechanical performance under these optimized processing conditions. However, it is still slightly lower than that of neat PP. All the parameters for preparing distinct welds are shown in Table 3.

Parameter	Unit	Value
Weld pressure	MPa	0.4
Vibration amplitude	mm	0.7
Meltdown	mm	0.8
Vibration frequency	Hz	240

Table 3: Welding parameters

2.3 Thermal treatment of the welds

After successful welding of the samples, they were annealed firstly in a heating cabinet, Binder GmbH, Germany, at distinct temperatures between 125 °C and 140 °C for pre-defined annealing time, which is above the crystallization onset temperature of PP from non-isothermal differential scanning calorimetry (DSC) measurement, Figure 1. In addition, Infrared radiation served as an in-line annealing technique, as is shown in Figure 2. Two quartz halogen infrared heaters, QHS-1000, Ceramicx, Ireland, with a power of 1000W and peak wavelength emissions of approximately 1 μm were installed on the welding machine. Its heating temperature can arrive a maximum temperature of about 2500°C. After the welding process was completed, the infrared heaters were activated immediately for annealing the weld. In order to guarantee the best heating effect and reduce the processing time, the distance between the sample and the heater was chosen as 75 mm for PP and 65 mm for PP-S after process optimization. Pre-test revealed that the temperature in the weld region reached the target temperature after approximate 10 minutes and thereafter remained constant. Therefore, the radiation periods of 10 and 20 minutes were chosen for pure PP. Due to the high thermal conductivity after adding nanoparticles, the treating time of PP-S was fixed at 15 minutes (mean value of 10 and 20 minutes).

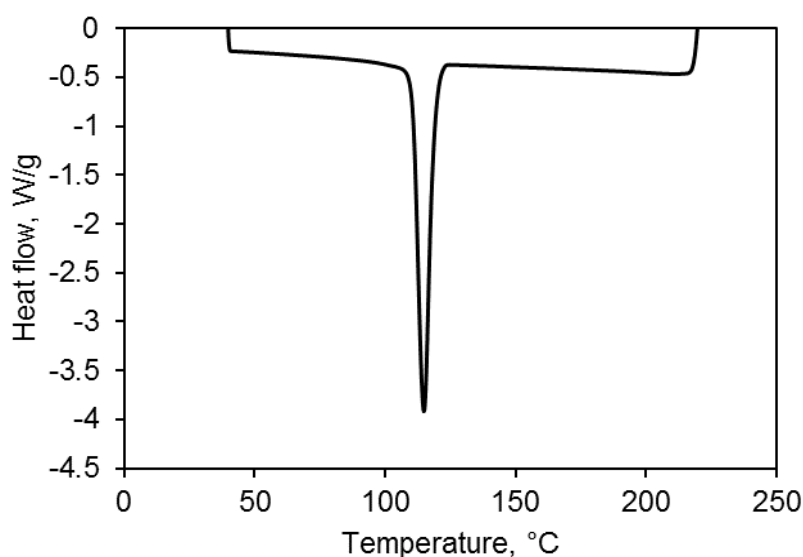


Figure 1: DSC cooling curve of neat PP

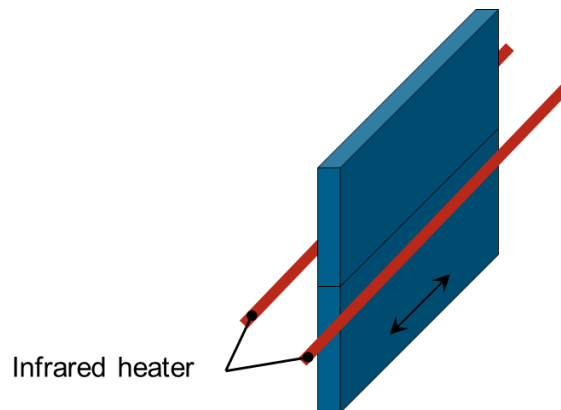


Figure 2: Schematic illustration of in-line infrared radiation technique on annealing the weld

2.4 Mechanical investigations

Tensile properties of the welds were measured on a universal tensile testing machine (Retroline, Zwick GmbH & Co. KG, Germany) at room temperature according to DIN EN ISO 527-2/1BB. The weld bead was not removed for determining the tensile properties. The crosshead speed was chosen as 1 mm/min and 50 mm/min for determining the Young's modulus and ultimate tensile strength, respectively. At least five samples of each material were tested for calculating the mean values of the mechanical properties.

2.5 Thermal properties

Thermal characteristics of the welds were analyzed by using differential scanning calorimetry (DSC), DSC Q20 apparatus, TA Instruments, USA. The sample was heated from 40 °C to 220 °C with a heating rate of 10 K/min and kept at this temperature for 5 minutes, in order to eliminate the residual stress and thermal histories. Afterwards, the sample was cooled down to 40 °C with a cooling rate of 10 K/min. The crystallinity of the materials was calculated by applying the following equation, Equation 1.

$$X_c = \Delta H_m / (w_p \cdot \Delta H_c) \quad (1)$$

Whereby, X_c is the degree of crystallinity, ΔH_m indicates the melt enthalpy from DSC measurement, w_p and ΔH_c are the weight content of PP within the nanocomposite and the theoretical 100% crystallized PP, which is taken as 209 J/g [13].

2.6 Morphological characterizations

Dispersion and distribution of nano-SiO₂ within the polymer matrix were analyzed by means of a focused ion beam (FIB, Altura 875 Dualbeam, FEI,

USA). Prior to the FIB analysis, the polymer sample surface was sputtered with a platinum layer in order to create electrically conductive surface for the analysis. These FIB images served as input data for determining the nanoparticle agglomerate size distribution by using Nikon NIS-Elements processing software. Firstly, these FIB-images were switched into black-white images in which nanoparticle agglomerates displayed white color. Afterwards, the area of the agglomerates can be calculated, from which equivalent diameter of the agglomerates can be determined.

3 RESULTS AND DISCUSSION

3.1 Micrograph of PP nanocomposite

As is well known, the quality of dispersion and distribution of nano-sized fillers within the polymer matrix play a dominant role on determining the properties of the nanocomposites, e.g. mechanical and electrical properties etc. Hence, dispersion and distribution of nanoparticles within the PP matrix was analyzed by using FIB, and the representative micrograph is shown in Figure 3 (left). As is shown, nano-sized silicon dioxide particles almost homogeneously disperse and distribute within the PP matrix. However, careful inspection of the micrograph reveals that agglomerates of SiO₂ nanoparticles up to 200 nm can be still observed in the nanocomposite. Quantitative analysis of the agglomerate size distribution by using Nikon NIS-Elements processing software reveals that approximate 99.7% of the agglomerates are smaller than 100 nm. More important, approximate 63% of the agglomerates exhibits a size up to 40 nm, which indicates an excellent dispersion of the nanoparticles within the PP matrix, Figure 3 (right).

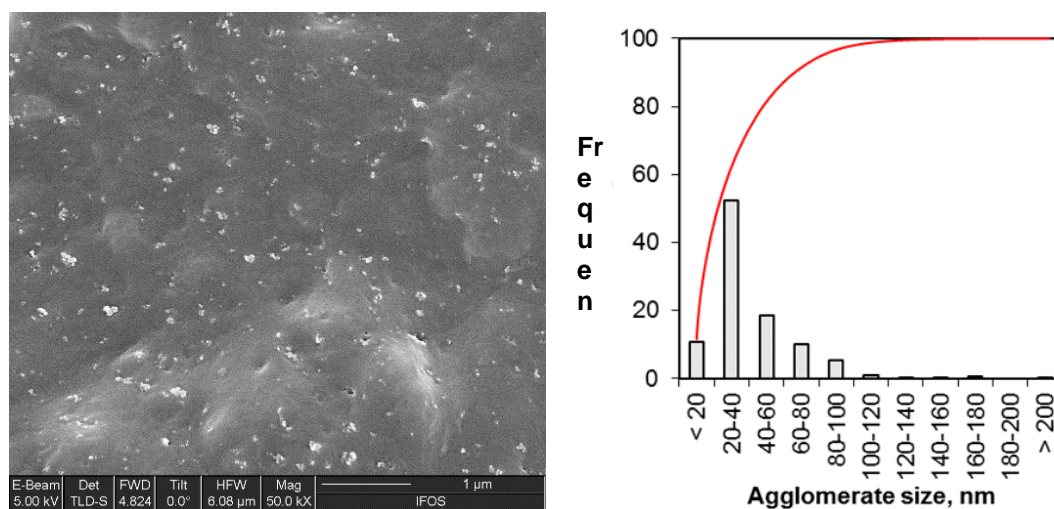


Figure 3: Dispersion and distribution of the nanoparticles within PP matrix.

Left: Micrograph, right: Agglomerate size distribution

3.2 Mechanical properties

Figure 4 shows the dependence of the mechanical properties of the weld on the annealing conditions by using a heating cabinet, i.e. temperature and duration of annealing. As can be seen, the annealing temperature and time exert obvious influence on the Young's modulus of the vibration weld. For both materials, i.e. virgin PP and PP nanocomposite, annealing of the weld in a heating cabinet leads to increasing modulus. Maximum tensile modulus is to be observed at 140°C with 72 h annealing time. Improvement of the modulus reaches 36% and 30% for PP and PP/nano-SiO₂ composite, respectively, compared to that of non-treated weld. Moreover, addition of nano-sized rigid particles into PP matrix leads to increasing modulus independent on the annealing parameters. With respect to the weld strength, similar trend can be also observed after annealing of the weld. The weld exhibits high weld strength after annealing process for both polymeric materials. More interestingly, unlike the decreased weld strength of neat PP after adding silicon dioxide nanoparticles without thermal treatment (reference material, Figure 4, right), nanocomposite shows slightly higher tensile strength after treatment at annealing temperatures above 135°C, Figure 4 (right).

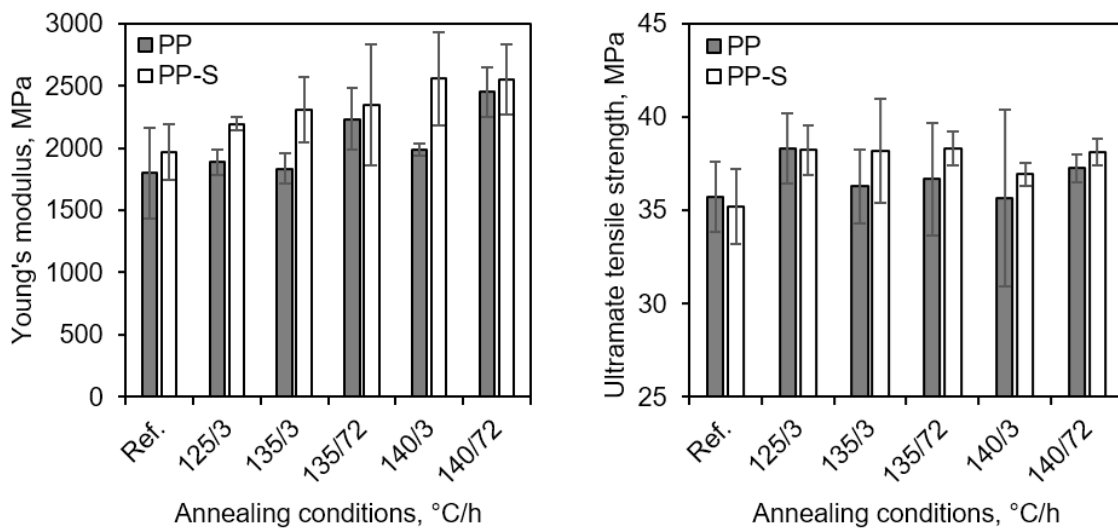


Figure 4: Dependence of Young's modulus and tensile strength of the welds under different annealing conditions in the heating cabinet.

Left: Young's modulus, right: Tensile strength

Considering the weld strength by using in-line infrared heating technique, it is revealed that high temperature and radiation time result in high stiffness and strength of the weld especially the weld strength, as is shown in Figure 5. The maximum improvement of the tensile strength of the welds is 6% and 12% for neat PP and PP nanocomposite, respectively, compared to that of the corresponding reference weld without any thermal treatment. In comparison to the thermal treatment in a heating cabinet, infrared annealing during the welding process provides attractive results of PP nanocomposite. It results in high weld strength at 140°C within significantly short treating time (72 h vs 15 min), Figure

4 (right) and 5 (right), which is much important for reducing the cyclic time for industrial production. More importantly, the negative influence of adding nanofillers on the weld strength without thermal treatment (cf. Figure 4, right, tensile strength of reference samples), which has been reported in early study of Bates et al [8] and our recent work [9, 14], was compensated by the annealing process. Silicon nanoparticle filled PP exhibits enhanced weld strength compared to that of neat PP weld without thermal treatment. The distinct effects of different thermal treatments on the mechanical properties of the weld can be attributed to the different initial phases of the annealing process regarding the thermal history of the weld. Annealing of the weld within a heating cabinet took place after complete cooling of the weld. This heat treatment leads to a cold crystallization with an associated increase in the degree of crystallization. In addition, internal stresses can be reduced. In contrast, the infrared heat treatment is started immediately after the oscillation movement has been stopped, i.e. when the weld seam has not yet crystallized. Therefore, in this case the material in the weld crystallizes quasi-isothermally.

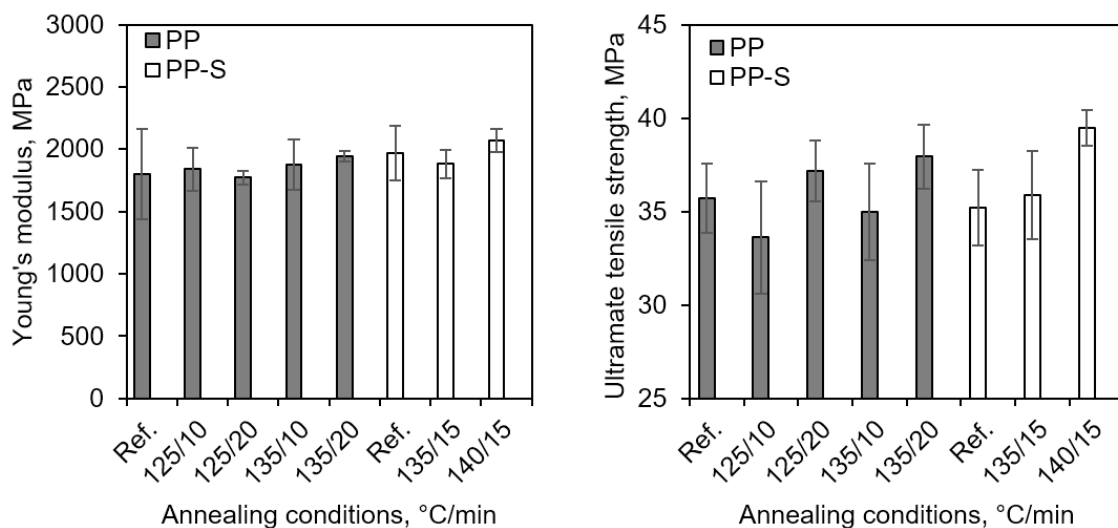


Figure 5: Effect of the annealing conditions on Young's modulus and tensile strength of weld strength by using in-line infrared radiation

Left: Young's modulus, right: Tensile strength

3.3 Thermal properties

Crystallinities of PP and PP nanocomposite in the weld area after annealing in the heating cabinet with distinct temperatures and periods are summarized in Table 4. It is noticed that the crystallinity of PP nanocomposite in the weld area without thermal treatment is obviously lower than the corresponding value of neat PP. This observation can be attributed on the one hand to the narrow weld PP after incorporating nanofillers that restricts the growth of spherulites in spite of the nucleation effect of tiny nanoparticles [9], on the other hand, to the high

thermal conductivity of the PP nanocomposite induced by fast cooling rate [15]. More interestingly, crystallinity of PP-S significantly increases after the annealing process. The maximum increase of the crystallinity is more than 15% at an annealing temperature of 140°C. In contrast, no obvious increment of crystallinity of PP can be observed after annealing in the heating cabinet.

Material	Annealing parameter °C/h	Crystallinity X_c %
PP	Ref.	56.8
	125/3	60.2
	135/3	47.7
	140/3	37.5
	135/72	53.6
	140/72	56.0
PP-S	Ref.	42.7
	125/3	47.0
	135/3	56.2
	140/3	58.8
	135/72	52.8
	140/72	58.0

Table 4: Degree of crystallinity of PP and PP-S under different annealing conditions within a heating cabinet

Taking into account the effect of thermal treatment by using infrared radiation on the crystallinity, it is found that in-line infrared radiation direct after the welding process also leads to an increase in the weld crystallinity of PP-S, similar to the influence of thermal treatment in the heating cabinet. In contrast, infrared treatment impedes the crystallization of neat PP in the weld region. The reduction of the crystallinity is more than 10% compared to that of untreated weld.

Material	Annealing parameter °C/h	Crystallinity X_c %
PP	Ref.	56.8
	125/10	45.4
	125/20	44.5
	135/10	47.4
	135/20	44.0
PP-S	Ref.	42.7
	135/15	44.3
	140/15	48.7

Table 5: Degree of crystallinity of pure PP and PP-S under different annealing conditions with infrared radiation

3.4 Discussion

Examinations of the effects of distinct thermal treatments on the mechanical and thermal properties of PP materials demonstrate that thermal treatment exerts positive effect on the weld quality, as mentioned above. In order to elucidate the mechanisms for improving mechanical properties after thermal treatment of the welds, the fracture pattern of the samples was examined. It is noted that the specimens fractured partly in the weld region and partly outside of the weld, which indicates that the weld strength attains the strength of the bulk material under optimized welding conditions selected (cf. Section 2.2). Plot of the weld strength as a function of the crystallinity in the weld area presents interesting results which are shown in Figure 6. As is seen, the weld strength exhibits quasi-enhancing trend with increasing crystallinity of neat PP after annealing within a heating cabinet. Similarly, the weld strength of PP-S enhances with increasing crystallinity up to 53% which remains constantly with further increment of crystallinity to 58%. Afterwards, a slight decrease of the weld strength at the crystallinity of 59% can be observed. However, it is still higher than the value at lowest crystallinity. Increasing tendency of the weld strength can be also observed on the infrared treated PP nanocomposite. In addition, virgin PP presents also increasing trend of the weld tensile strength above a crystallinity of 45%. It can be therefore concluded that crystallinity of PP materials in the weld area exerts obvious positive influence on the weld quality, especially for PP/SiO₂ nanocomposite. Because of the narrow weld region and fast cooling rate due to high thermal conductivity after adding nanoparticles, PP-S displays lower crystallinity in the weld area after vibration welding. However, post-processing of the PP-S weld by using thermal approaches results in obviously enhancing crystallinity. This increasing crystallinity is responsible for the high weld quality after thermal treatment which is coincident with the results reported in early studies that high crystallinity improves the mechanical due to enhancing load-bearing capacity [16, 17]. Not only the crystallinity, but also the size of the spherulite in the weld region can affect the mechanical properties of the weld. It is commonly accepted that increasing spherulite size also increases the mechanical properties of PP [17]. It is therefore believed that the high weld strength of neat PP below the degree of crystallinity of 45% after annealing with infrared radiation might be caused by the large spherulites in the weld region in spite of the relative low crystallinity.

However, this does not fully explain the difference between the different annealing processes. Presumably, the near-surface internal stresses in the component can already be effectively reduced by infrared annealing. In addition, it might be possible that the geometric notch caused by the weld, which is always a weak point, will be defused by the infrared heating. This must be clarified in further investigations.

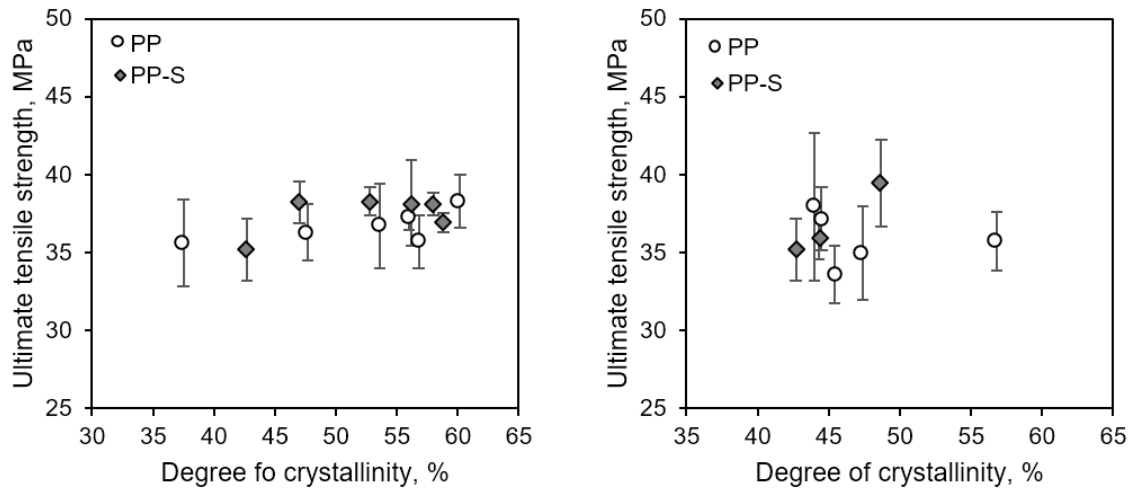


Figure 6: Dependence of the weld strength on the weld crystallinity with distinct thermal treating approaches

Left: heating cabinet, right: Infrared radiation

4 CONCLUSIONS

The effects of different annealing processes on the weld properties of PP materials were systematically studied in this work. Following conclusions can be drawn:

- Addition of silicon dioxide nanopartilces slightly impairs the weld strength of PP without post-processing annealing.
- Thermal treatment of the weld leads to obviously enhancing young's modulus and tensile strength.
- For infrared treated welds, the strength of the PP/SiO₂-nanocomposite improves with increasing degree of crystallinity.
- Even if infrared radiation might not be able to fully penetrate to the center of the weld is more efficient technique for annealing the weld compared to the thermal treatment in a heating cabinet.

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