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금속 열순환 성형에서 수지의 점도와 윤활유가 유리 섬유 강화 고분자의 기계적 물성과 표면에 미치는 영향

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Effect of Resin Viscosity and Lubricants on Surface and Mechanical Properties of Glass Fiber Reinforced Polymer in Rapid Heat Cycle Molding

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Abstract: Conventional injection molding (CIM) and rapid heat cycle molding (RHCM) methods are used to prepare ABS and (acrylonitrile-butadiene-styrene)/glass fiber copolymer (ABS/GF) plastic parts. Pentaerythritol stearate (PETS) and silicon particles are chosen as lubricants. Effect of resin viscosity and lubricants on properties of plastic parts was investigated. The results show that resin viscosity has some effect on surface quality of plastic parts in CIM process but no effect in RHCM process. ABS/GF plastic parts molded in RHCM process exhibit higher tensile, higher flexural strength and lower impact strength. Silicon is more effective in RHCM process than in CIM process.

Keywords: injection molding, rapid heat cycle molding, properties, viscosity, lubricants.

Introduction

Glass fiber reinforced polymer has been of great interest to researchers for decades.[1,2] Conventional injection molding (CIM) method is an important technology to fabricate complex shaped plastic parts. However, mold temperature is controlled by circulating coolant through the cooling channels in CIM. In order to shorten the cooling stage which usually occupies more than half of molding cycle, the set mold temperature is often lower than the ejection temperature of plastic parts.[3] The rather cold mold makes resin melt to freeze in advance during filling stage, which brings a series of appearance defects of plastic parts,[4] such as weld line, flow marks, crazing defects and floating fibers, especially for glass fiber reinforced plastic parts. These defects are usually removed by using second processing technology, such as polishing, electroplating and spraying, which aggravates environmental pollution, waste of energy and increasing of cost.

Rapid heat cycle molding (RHCM) is an advanced injection molding technology and it can overcome inherent defects of conventional injection molding parts. A dynamic mold temperature control method is introduced into RHCM process. The mold cavity is rapidly heated up to the glass transition temperature of polymer before filling stage. During filling and packing stage, the mold should be always kept at high temperature in order to improve the flow ability of the polymer melt. At the end of packing stage, the mold cavity is cooled down quickly so as to freeze the polymer melt for demolding. And then the mold will be heated again for the next injection cycle. Since the mold cavity temperature is always kept at a high level during filling and packing stage, the flow ability of melt is significantly improved and the premature freezing of polymer melt during filling stage is avoided. Products made in RHCM process are usually put into practical use without painting. So RHCM process has a broad development prospect due to its environmental protection and low cost.

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Recently, the researches of RHCM process are almost concentrated on heating methods of mold[5-16] and the effect of processing parameters on surface and mechanical properties of plastic parts.[17-23] Xie[17] investigated the influence of processing parameters on weld line mechanical properties of polypropylene (PP) in RHCM process. It was found that the influencing significance order of processing parameters from strong to weak were mold temperature, melt temperature, injection speed, ejection temperature, packing pressure and injection pressure. The V notch size in the specimens' middle part was larger and deeper than that in the edge. Wang[18] researched the reduction of sink mark and warpage of the molded part in RHCM process. The external gas assisted packing was also proposed to reduce the sink mark in RHCM process and the warpage of plastic parts was reduced effectively. Liu[19] investigated the influence of mold temperature on shrinkage of plastic part in RHCM process. Results revealed that the shrinkage of RHCM part was reduced obviously compared with conventional injection plastic part. In addition to mold temperature, both packing pressure and packing time also had important influences on the shrinkage of plastic part. Wang[20] found RHCM process can greatly increase the surface gloss of fiber-reinforced plastics. Li[21] studied the influence of mold temperature on tensile strength of plastic parts in RHCM process. The relationship between dynamic mold temperature and tensile strength of plastic parts was demonstrated. Zhang[22] investigated the effect of mold temperature on surface quality and mechanical properties of ABS/PMMA/nano-$CaCO_3$ in RHCM process. The results revealed surface quality of ABS/PMMA/nano-$CaCO_3$ parts was improved significantly by increasing mold cavity temperature in RHCM process. And weld line was eliminated in RHCM process. Wang[23] researched the effect of mold temperature on crystal structures, morphology of polypropylene and surface quality of plastic parts. The crystallization status in surface layer, subsurface layer and core was studied under different mold temperature in RHCM process.

In a word, the replication ability of resin matrix is the key to determine final surface quality of plastic part.[24,25] Mold temperature is one of the most important parameters which influence the replication ability of resin melt in injection process. The high mold temperature can improve the surface quality of plastic parts by improving the replication ability of resin matrix in RHCM process. However, mold temperature is not the only parameter related to the replication ability of resin matrix. The viscosity of resin matrix is another parameter related to the replication ability of resin matrix. It is of great significance to clarify the relationship between viscosity of resin matrix and surface quality of plastic parts in RHCM process. However there has been little research about the impact of viscosity on surface quality of plastic parts in RHCM process.

In addition, additives are essential in the process of injection molding. The influence of additives on the properties of plastic parts cannot be ignored. Compared with CIM process, RHCM technology involves more processing parameters and more complex process control. The high temperature of cavity wall in filling stage changes the filling process of resin melt and the RHCM forming mechanism is quite different from CIM's. Whether the effect of additives in RHCM process is the same as it in CIM process is not clear.

In this paper, SAN (styrene acrylonitrile) was incorporated into ABS based on twin screw compounding in order to decrease the viscosity of ABS and then the LV-ABS (low viscosity ABS) was prepared. Then the effect of rein melt's viscosity on surface and mechanical properties of polymer plastics in RHCM and CIM processes was investigated. Lubricant is one of the most important additives in glass fiber reinforced polymers. Pentaerythritol stearate (PETS) and silicon particles were chosen as lubricants for ABS and ABS/GF in our study. Then the effect of PETS and silicon on properties of ABS and ABS/GF plastic parts in RHCM and CIM process was studied.

Experimental

Materials. ABS resin (GP22) and SAN resin (NF 2200) were purchased from BASF Company and Taiwan Formosa chemicals & fibre corporation, respectively. Silicon particles (KJ-A01) which was a kind of ultra-high molecular weight lubricants were obtained from Zhejiang Jiande Kaijie plastic toughening materials Co. And its information is illustrated in Table 1. PETS was provided by Guangzhou Chuangxin chemical Technology Co., Ltd. And the glass fibers with a length of 3 mm were provided by Zhejiang Tongxiang Jushi Group.

Sample Preparation. Preparation of LV-ABS: ABS is synthesized from three monomers of acrylonitrile, butadiene and styrene. It is an amorphous material with two phases, one is the continuous phase of styrene acrylonitrile (SAN), and the other is the dispersed phase of polybutadiene rubber. Acrylonitrile styrene copolymer has low viscosity and excellent flow ability. The viscosity of ABS can be adjusted by changing the proportion of SAN in ABS. Both ABS and SAN were predried in an air circulating oven at 80 ^{o}C for 12 h to remove all

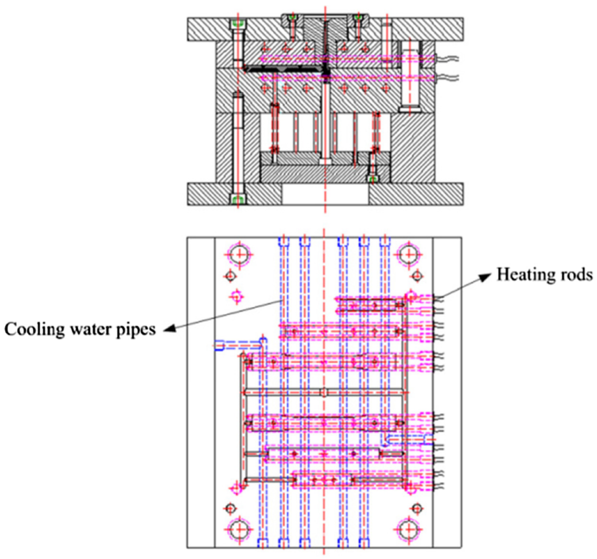

Figure 1. Drawing of electric heating mold.

the moisture. Low viscosity ABS (LV-ABS) pellets were obtained by blending ABS and SAN (weight ratio: 90/10) in a co-rotating twin screw extruder with a screw diameter of 21 mm and length to diameter ratio of 36. Temperature profile of 205-225-235-220-180 °C was set from the hopper to the die. The feed rate was kept at 3.6 kg/h and the screw speed was set at 120 rpm. The melt flow behavior of ABS was characterized by melt flow index (MFI). The MFI testing was carried out in a XNR-400 melt flow index tester at 230 °C and 3.8 Kg. The melt flow index (MFI) of ABS was 2.6 g/10 min and MFI of LV-ABS is 3.2 g/10 min.

Preparation of ABS and ABS/GF Plastic Parts: An electric heating RHCM system was developed in our laboratory. Two heating rods with the power of 15 W/cm^2 were used for each sample in order to heat the mold quickly. Cooling water pipes were arranged vertical to heating rods as illustrated in Figure 1. Six specimens according to ASTM standard specifications were molded each injection cycle. And the tensile specimen, flexural specimen and impact specimen with one gate were used in this paper for characterization. The RHCM system was built on the platform of MA3200 injection molding machine. The diagram of RHCM process is showed in Figure 2. The mold temperature was kept at 115 °C during filling stage in RHCM process. Melt temperature, injection time, packing pressure and packing time were kept constant.

All the materials were dried once again in an air circulating oven at 80 °C for 12 h. The materials with different formulas according to Table 1 were premixing in a container. Then specimens were molded using RHCM process or CIM process according to Table 1.

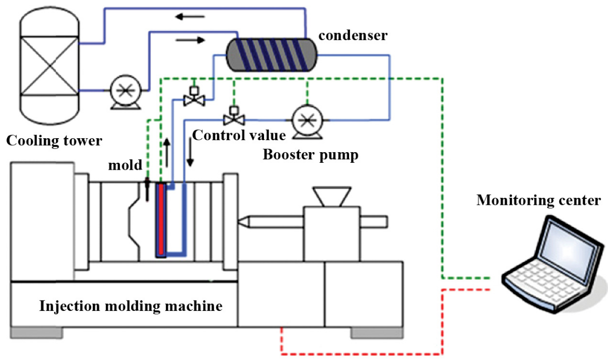

Figure 2. The diagram of RHCM process.

Table 1. Formations and Molding Methods of Composites

Code	Formations	Glass fibers (%)	Molding methods
1	ABS	0	CIM
2	ABS	0	RHCM
3	ABS	30	CIM
4	ABS	30	RHCM
5	LV-ABS	0	CIM
6	LV-ABS	0	RHCM
7	LV-ABS	30	CIM
8	LV-ABS	30	RHCM
9	ABS/silicon: 99/1	0	CIM
10	ABS/silicon: 99/1	0	RHCM
11	ABS/silicon: 99/1	30	CIM
12	ABS/silicon: 99/1	30	RHCM
13	ABS/PETS: 99/1	0	CIM
14	ABS/PETS: 99/1	0	RHCM
15	ABS/PETS: 99/1	30	CIM
16	ABS/PETS: 99/1	30	RHCM

CIM: conventional injection molding; RHCM: rapid heat cycle molding.

Characterization. All tensile specimens were dumb-bell type suitable to ASTM standard specifications. Tensile tests were carried out on a CMT 4204 20KN universal testing machine (maximum load: 20 KN) at room temperature according to ASTM D638.[26] The crosshead speed was kept at 5 mm/min. And flexural tests were also carried out on a CMT 4204 20KN universal testing machine (maximum load: 20 KN) at room temperature according to ASTM D790.[27] The crosshead speed was kept at 2.0 mm/min and the support span is 16 times specimen depth. The Izod impact strength tests were conducted on a XC-5.5D impact tester at room temperature according to ASTM D256.[28] At least five samples were performed for each property and the average values were calculated and used for this study.

And a JFL-BZ60° gloss meter, produced by Tianjin jinfulun Inc., was utilized to measure the gloss of the part surface. The surfaces of injection specimens were observed by a JEOL 6610-LV SEM. Polymer specimens were coated with gold in an automatic sputter coater (E-1010 ion sputter) before the SEM observations.

Results and Discussion

Effect of Viscosity and Molding Methods. Surface Quality: The surface quality of injection plastic parts is related to the replication ability of resin melt and the surface quality of mold cavity. When replication ability of resin melt is better and surface of mold cavity is glossy and smooth, plastic parts with better surface quality can be obtained in injection process. Since the surface quality of mold cavity is certain in actual production, improving the replication ability of the resin melt becomes the key to improve surface quality of injection plastic parts. The replication ability of resin melt is closely related to resin melt's viscosity and mold temperature in filling stage, especially the mold temperature.[29]

The gloss of various injection plastic parts was illustrated in Figure 3. In order to investigate the influence of viscosity on surface quality of plastic part, we compared the gloss of ABS plastic part and LV-ABS plastic part in both CIM and RHCM

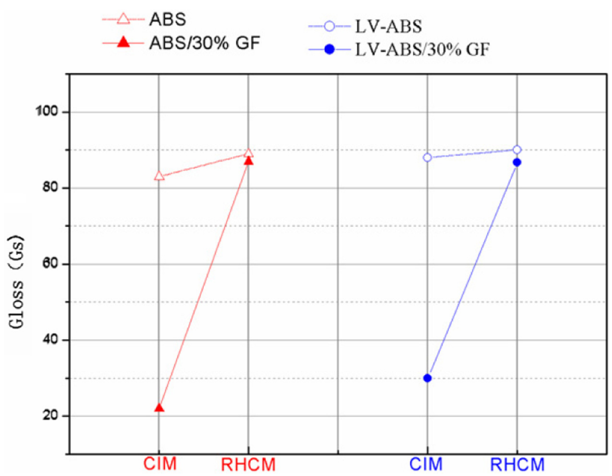

Figure 3. Gloss of various samples by CIM and RHCM methods.

process firstly. It can be found that gloss of LV-ABS part is a little higher than that of ABS part when CIM method is used. In CIM process, the mold temperature is relatively lower than the ejection temperature of plastic parts and the temperature of resin melt decreases quickly as soon as it contacts mold cavity wall. A premature frozen layer is apt to be formed during filling process. There is not enough time for resin melt to replicate the mold cavity surface completely. In limited time, resin melt with low viscosity can replicate the surface of mold cavity better. From the view of molecular motion, the viscosity of polymer melt is determined by two factors: the free volume in polymer melts and the entanglement between the molecules. The free volume is the void not occupied by polymer chains in polymer matrix. And all the factors that can increase the free volume can reduce the viscosity of polymer. The entanglement between polymer chains increases the internal friction. Any factors that cause the entanglement density can make the movement of polymer chains difficult. Low viscosity means that polymer chains are easy to move. That is polymer chains can replicate the mold cavity wall much more easily. Therefore, LV-ABS exhibited better replication ability than ABS in CIM process. So there is some effect of viscosity on surface quality of ABS plastic part in CIM process.

However, the gloss of ABS part is equal to the gloss of LV-ABS part when RHCM method is used. The mold temperature is above the glass transition temperature of resin melt in RHCM process. The temperature of resin melt is always kept at a relatively high level during filling stage. The viscosity of both ABS and LV-ABS resin melts is lower during filling stage just because the viscosity of polymer is decreased significantly with the increase of temperature. Furthermore, premature frozen layer is avoided in RHCM process. There is enough time for resin melt to replicate the mold cavity surface completely. Therefore, it can be seen that there is little influence of resin viscosity on surface quality of plastic part and mold temperature is the critical factor influencing the surface quality of plastic parts in RHCM process.

Similarly, the gloss of LV-ABS/GF part is a little higher than that of ABS/GF part by using CIM method and the gloss of ABS/GF part is equal to the gloss of LV-ABS/GF part in RHCM process. The reason is the same as ABS parts without GFs.

However, when GFs were incorporated into ABS resin, the glosses of both ABS/GF and LV-ABS/GF parts decease significantly in CIM process as shown in Figure 3. Floating fiber is the culprit to destroy the surface quality of plastic part. Fig-

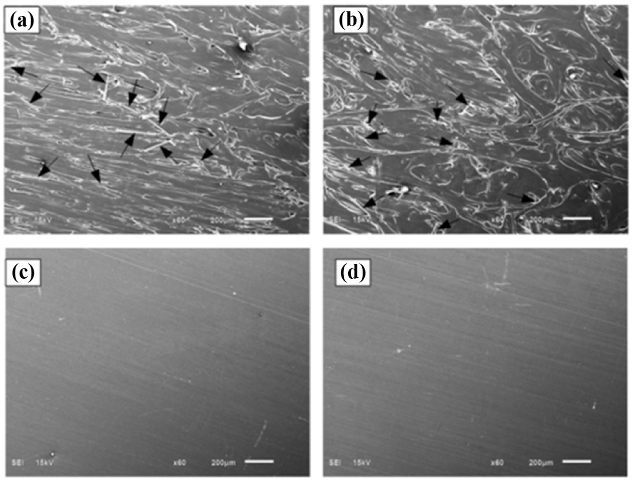

Figure 4. SEM images of sample surface: (a) ABS/30%GF CIM samples; (b) LV-ABS/30%GF CIM samples; (c) ABS/30%GF RHCM samples; (d) LV-ABS/30%GF RHCM samples. Scale bar: 200 μm.

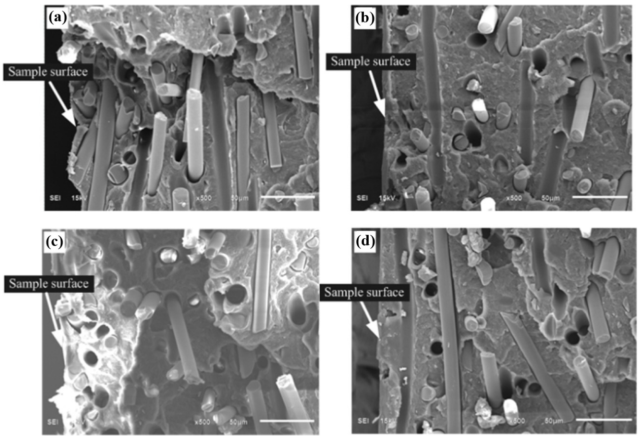

Figure 5. SEM images of fracture surfaces by Izod impact tests: (a) ABS/30%GF CIM samples; (b)ABS/30%GF RHCM samples; (c) LV-ABS /30%GF CIM samples; (d) LV-ABS /30%GF RHCM samples. Scale bar: 50 μm.

ure 4(a), (b) show the SEM images of plastic part surfaces in CIM process and Figure 4(c), (d) show the SEM images of plastic parts surfaces in RHCM process. The surfaces of both ABS/GF and LV-ABS/GF in CIM process are much worse than that in RHCM process. And many floating fibers can be found in Figure 4(a), (b) as marked by black arrows. Figure 5 illustrates SEM micrographs of fracture surfaces of composites by Izod impact tests. Floating fibers can be found in Figure 5(a) and 5(c) and their surfaces are rough. However the surfaces are smooth and there are no fibers revealed out of surface in Figure 5(b) and 5(d). Floating fibers were eliminated in RHCM process.

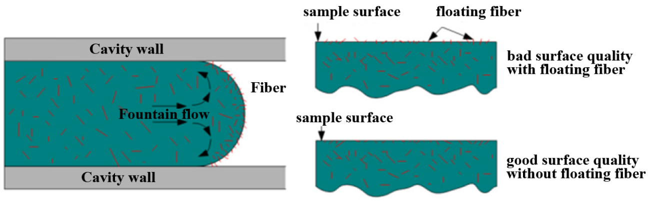

Figure 6. The diagrammatic sketch of floating fiber.

The interface layer between GF and resin matrix will be destroyed by interior shear stress of resin melt during filling stage. When the interfacial adhesion decreased to a certain extent, GF and resin matrix will be separated because they are incompatible and their density is different. Moreover, the resin melt presents the fountain flow behavior during filling stage as illustrated in Figure 6. The GF will be accumulated in the melt front due to its low density. In CIM process, premature frozen layer makes the melt unable to wrap the GFs fully and then the floating fibers formed. The quality of plastic part surface becomes worse. The glosses of ABS/GF and LV-ABS/GF are below 30 Gs as shown in Figure 3. However, the frozen layer will be avoided in RHCM process and the resin melt can perfectly wrap GFs. Thus the floating fibers are eliminated. The surface glosses of ABS/GF and LV-ABS/GF parts improved greatly in RHCM process compared with CIM process.

The gloss of ABS/GF parts and ABS parts stabilized at the same level. It can be found that the bad influence of GF on surface quality of plastic parts is eliminated totally in RHCM process.

Mechanical Properties: Table 2 shows the mechanical properties of ABS and ABS/GF composites. It clearly demonstrates that tensile strength as well as flexural strength of ABS in RHCM process is lower than that in CIM process. And LV-ABS is similar with ABS for tensile strength and flexural strength. The velocity of resin melt near the cavity wall is zero due to the premature frozen layer in CIM process. Therefore the velocity gradient and shear rate inside the resin melt will be increased. Polymer chains are easily orientated with high shear rate in CIM process.

However shear rate is lower in RHCM process than that in CIM process because the frozen layer is eliminated in RHCM process. In addition, the melt temperature decreases slowly and severe thermal motion of polymer chains leads to the disorientation in RHCM process. Both lower shear rate and full disorientation give rise to lower orientation degree of polymer chains in RHCM process. The polymer chains' orientation is beneficial to improving strength of plastic parts. Therefore

Table 2. Mechanical Properties of ABS and LV-ABS Composites

Mechanical properties	Glass fiber content	ABS			LV-ABS		
		CIM	RHCM	Increment	CIM	RHCM	Increment
Tensile strength	0	37.3	36.7	-1.61%	37.9	37.6	-0.79%
	30	38.1	38.5	1.05%	38.2	39.3	2.88%
Flexural strength	0	68.1	62.8	-7.78%	68.2	67.6	-0.88%
	30	69.9	70.4	0.72%	69.8	72.8	4.30%
Izod impact strenth	0	166.9	181	8.45%	157.4	162.3	3.11%
	30	63.5	57.7	-9.13%	60	57.9	-3.50%

Postscript: Positive value represents increasing and negative value represents reducing.

both tensile and flexural strength of ABS in CIM process is higher than that in RHCM process. LV-ABS is the same with ABS for tensile and flexural strength.

When GFs are added into ABS resin, the tensile and flexural strength of RHCM parts are a little higher than that of CIM parts. This results contrast with ABS parts discussed above. The interfacial layer between GF and resin melt will be destroyed by interior friction of resin melt. In CIM process, high shear rate makes the interfacial adhesion worse and worse. As we all known, interfacial adhesion between fibers and resin matrix is one of the most important factors which influence the strength of fiber reinforced polymers. Therefore, the strengths of ABS/GF and LV-ABS/GF composites in RHCM process are a little higher than that in CIM process.

Izod impact strength is closely related to the internal stress of plastic part. The smaller the internal stress is the higher the impact strength is. Orientation stress and thermal stress are two kinds of internal stresses created in injection process. Orientation stress and thermal stress are attributed to the orientation of polymer chains and the temperature gradient of plastic parts respectively. As discussed in the previous paragraph, the orientation degree of polymer chains is reduced in RHCM process. Therefore the orientation stress of RHCM plastic parts is reduced. The premature frozen layer will be eliminated in RHCM process. This illustrates that all of the melt (skin and core) will maintain high temperature during the filling stage. Temperature gradient from core to skin will also be reduced. Apparently, thermal stress created by temperature gradient is decreased to a low level. The reduction of orientation stress and thermal stress will give rise to the increase of Izod impact strength as shown in Table 2. This is similar with ABS/PMMA blends our research group studied before.[22]

Comparing with ABS, the variation of Izod impact strength for ABS/GF in RHCM and CIM process is opposite. As we discussed before, resin phase can flow freely in the mold and wrapped GFs in RHCM process. This will cause uneven distribution of GFs in the plastic parts. Most of GFs will be aggregated in the core. The incorporation of GFs will deprave the toughness of plastic parts. The more the GFs are, the lower the Izod impact strength of plastic part is. Most of GFs in the core significantly decrease the toughness of plastic part. The inhomogeneity of plastic part causes reduction of Izod impact strength. On the contrary, the reduction of internal stress will increase the Izod impact strength. With the two influences, Izod impact strength of ABS/GF is decreased slightly in RHCM process. For mechanical properties, the effect of molding method on ABS and LV-ABS is almost the same.

Effect of Lubricants in CIM and RHCM Process. Pentaerythritol stearate (PETS) and silicon are both lubricants usually used in polymer molding process. In this paper, we investigate the effect of PETS and silicon on properties of ABS/GF composites in both RHCM and CIM process. The contents of PETS and silicon are both 1 wt%. Figure 7 plots the effect of PETS and silicon on tensile strength of ABS and ABS/GF composites. ABS plastic parts' tensile strength is decreased by adding 1 wt% PETS in both CIM and RHCM process, so are ABS/GF plastic parts. PETS, acting as internal lubricant, can weaken the cohesive force between polymer chains. It makes polymer chains to slide and rotate easily. This means that PETS has the function of plasticizer. Therefore, tensile strengths of both ABS and ABS/GF decrease with only 1wt% PETS. The plasticization effect of PETS in CIM process and RHCM process is similar.

Similar with PETS, silicon is also a kind of inner lubricant used in polymer. For ABS resin, the tensile strength is decreased by adding silicon due to its lubrication effect in both CIM and RHCM process. However, the tensile strength of ABS/silicon/GF composites is increased compared with ABS/

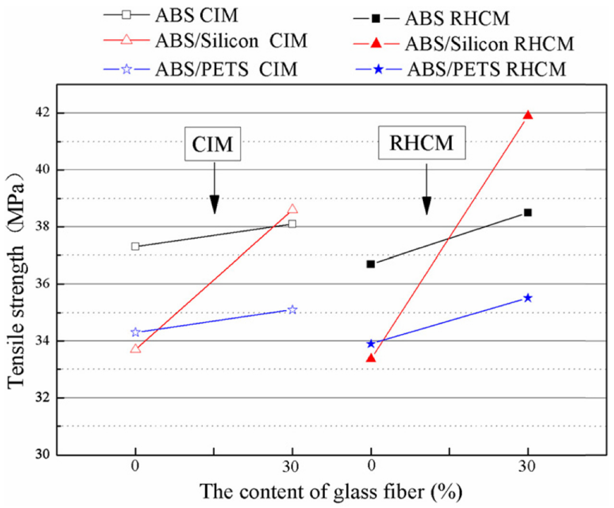

Figure 7. Tensile strength of ABS and ABS/GF composites.

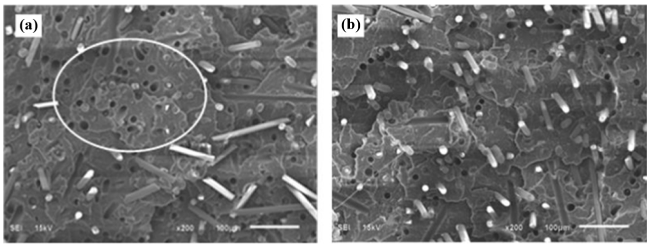

Figure 8. SEM images of tensile fracture surfaces in RHCM process: (a)ABS/GF; (b) ABS/silicon/GF. Scale bar: 100 μm.

GF composites in CIM and RHCM process. Silicon particle has a core-shell structure with an inorganic core of silicon and a polymer shell of polysiloxane. The chemical composition of GF is silicate which is similar with silicon. The interaction force of silicon particle's core and GF is strong. And the organic shell of silicon particle has good compatibility with polymer matrix. Thus the silicon particle holds the polymer matrix and GFs as a bridge. And therefore, the interfacial adhesion between polymer and GF is strengthened. So the tensile strength of ABS/GF composites is increased by adding silicon.

Figure 8 shows the tensile fracture surfaces in RHCM process. Figure 8(a) shows the fracture surface of ABS/GF without silicon and Figure 8(b) is the fracture surface of ABS/GF with silicon. There are many holes in Figure 8(a) marked by white circle. These holes are created by GFs when they are pulling out of resin matrix. This phenomenon proved that the interfacial adhesion between GFs and the resin matrix is worse. However, the holds are decreased in Figure 8(b) as silicon is incorporated into the materials. Silicon can improve the bonding force between GFs and resin matrix. In addition, silicon is more effective in enhancing the bonding force between GFs and resin matrix in RHCM process than in CIM process. Figure 7 illustrates that tensile strength of ABS/silicon/GF in RHCM process is 8% higher than that in CIM process. This may be attributed to the uniform distribution of silicon in RHCM process. In RHCM process, the shear rate is not too high and the flowing of resin melt is stable. The lubricant can be well distributed on the surface of GFs. The flexural strength of composites is similar to that of tensile strength as illustrated in Figure 9.

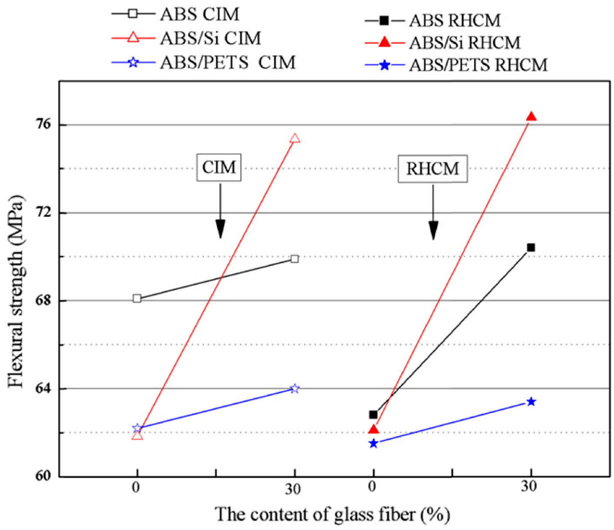

Figure 9. Flexural strength of ABS and ABS/GF composites.

Conclusions

LV-ABS is prepared by blending ABS and SAN in this paper. ABS, LV-ABS, ABS/GF and LV-ABS/GF parts were molded by CIM and RHCM process. The effects of viscosity and lubricants on surface quality and mechanical properties of plastic parts are investigated.

In RHCM process, mold temperature is the critical factor influencing the surface quality of plastic parts and there is little influence of resin viscosity on surface quality of plastic parts. However, in CIM process, the resin viscosity has a little effect on the surface quality and lower viscosity can increase the surface gloss of plastic parts to some extent. The gloss of ABS/GF plastic part deceases significantly compared with ABS plastic part in CIM process. Floating fiber is the culprit to destroy the surface quality of plastic parts and it can be eliminated by using RHCM process. The gloss of ABS/GF composite is significantly improved in RHCM process.

ABS plastic part in RHCM process exhibits lower tensile and flexural strength than that in CIM process due to its lower orientation of polymer chains. But its Izod impact strength in RHCM process is higher than that in CIM process owing to the reduction of orientation stress and thermal stress. However, when GFs are added into ABS matrix, the variation of strength presents completely opposite rule. ABS/GF plastic part in RHCM process exhibits higher tensile and flexural strength than that in CIM process. This is because high shear rate makes the interfacial adhesion between GFs and resin matrix worse and worse in CIM process. The inhomogeneity of GFs in resin matrix causes reduction of Izod impact strength of ABS/GF in RHCM process. LV-ABS is the same with ABS for mechanical properties.

PETS and silicon both exhibit lubrication effect in polymer matrix. And their incorporation makes strength of ABS deceasing in both CIM and RHCM process. However, for ABS/GF composites, the interfacial adhesion between polymer and GF is strengthened by silicon and therefore the strength of ABS/GF is improved by adding only 1 wt% silicon. Silicon is a kind of lubricants which plays the role of coupling agent. And silicon is more effective in enhancing the bonding force between GFs and resin matrix in RHCM process than in CIM process.

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