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Study of Mechanical Property and Cellular structure Based on the Controllable Crosslinking Polyethylene Foaming Materials

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Abstract. In this paper, the controllable crosslinking polyethylene foaming materials were established though adding foaming agent additives zinc stearate firstly. Then, the density, expansion ratio, cellular structure, gel content and compression strength of crosslinking polyethylene foaming materials were studied by changing zinc stearate content. Furtherly, those properties were researched after added nucleating agent. Experimental results showed that zinc stearate could facilitate foaming reaction and broad foaming window. Density, compression strength and gel content was decreased with zinc stearate content increased, and the expansion ratio increased with zinc stearate content increased. In addition, the compression strength was improved and cellular structure became uniform after adding nucleating agent. Generally, this material not only has the controllable property, but also has the higher mechanical property and more uniform structure.

1. Introduction

Because of the cellular structure and lightweight advantages, polyethylene foaming materials are widely employed in different and diverse applications, in fields like thermal insulation, cushioning or packaging, polyethylene foaming materials are currently necessary materials^[1-4]. When used for these purposes, mechanical properties and cellular structure are usually the most important properties which need considered, where the plastic matrix and gas diffusion from the cells are the fundamental determinant for those properties^[5]. While, gas diffusion path could variate with polyethylene modify, for instance, by introducing crosslinks between the polymer chains, a network is formed which inhibit the gas release freely. In addition, network structure also improve heat and chemical resistance, stress cracking and shrinking^[6].

Presently, there are several methods for providing network formation in polyethylene foaming material via a crosslinking reaction: with peroxides, by use of radiation and by the use of silanes^[7-9]. Because the convenient and cheap of chemical initiator, chemical crosslinking was the more widely using method than other crosslinking method^[10]. Masayuki et al. used peroxide crosslinking liner low density polyethylene as addition to reduce heterogeneous deformation during foaming process, and finally a foam material with a uniform cell size distribution was obtained^[11]. The investigation demonstrated that chemical crosslinking could improve polyethylene mechanical property and reduce cell size.



Due to add chemical crosslinking agent, the obtaining crosslinking polyethylene foaming material have better mechanical property than polyethylene foaming material, but its expansion ratio was decreased, which is due to the enhancement of the elastic modulus^[12]. In addition, the foaming window was fixed and narrowed in this kind of chemical crosslinking polyethylene foaming system (usually, crosslinking reaction occurred in the front and foaming reaction occurred in the after)^[12]. A broad foaming window facilitates foam processing and it is a useful way to evaluate the foamability of polymers, while, a narrow foaming window tends to make higher demands on the foaming system, particularly on the temperature control system^[13].

In order to improve the expansion ratio, keep good mechanical property and broad foaming window, a controllable crosslinking polyethylene foaming materials were designed in this paper, in which, azodicarbonamide (AC) as the foaming agent, 2,5-dimethyl-2,5-double (tert-butyl peroxide) hexane (DBPH) as the crosslinking agent and zinc stearate (ZnSr) as the foaming agent addition that could facilitate foaming agent decomposition. Rubber Process Analyzer (RPA) was used for the in situ measurement of variation of viscosity and pressure after different amount of ZnSr added. Density and expansion ratio were also tested and analyzed. SEM was used to characterize the cellular structures. Compression strength as the typically mechanical property was also analyzed, especially in comparing with polyethylene foaming materials.

2 Experimental

2.1 Materials

Linear Low Density Polyethylene (LLDPE) DMDA7042 was purchased from Shenhua Baotou Co., Ltd., with melt index 2.6 g/10min and density 0.918 g/cm³. 2,5-dimethyl-2,5-double (tert-butyl peroxide) hexane (DBPH) from Arkema Co., Ltd was used as the crosslinking agent, it was purchased from Aladdin Chemistry Co., Ltd. Azodicarbonamide (AC) and zinc stearate (ZnSr) were employed as the chemical foaming agent and foaming agent additive, respectively, they were purchased by J&K Chemical. Talcum powder was used as the uncleaning agent, which was purchased from Sinopharm Chemical Reagent Beijing Co., Ltd.

2.2 Samples preparation

HAAKE mixture: in this experiment, there were four groups of samples, they were all used 7042 as the matrix resin. The first group of samples' mass ration of 7042, DBPH and AC were maintained a consist value, which is 100:1:3, and the amount of ZnSr were 0, 0.1, 0.2, 0.4, 0.8 and 1.6, respectively. The second group of samples' mass ration of 7042 and DBPH were maintained a certain value, which is 100:1, and the amount of ZnSr were 0, 0.1, 0.2, 0.4, 0.8 and 1.6, respectively. The third group of samples' mass ration of 7042 and AC were maintained a fixed value, which is 100:3, and the amount of ZnSr were 0, 0.1, 0.2, 0.4, 0.8 and 1.6, respectively. And the four group of samples' mass ration of 7042, DBPH, AC and talcum powder were maintained a fixed value, which is 100:1:3:1, and the amount of ZnSr were 0, 0.1, 0.2, 0.4, 0.8 and 1.6, respectively. Then mixed polyethylene resin and those chemical agents, finally, put the obtained mixture into twin screw extruder, the temperature was set at 130°C and rotation speed was 110r/min. In order to distinguish these haake mixture clearly, their sample code was listed in table1.

Table 1. Sample code list

Serial number	Mixture formula	Sample code
1	7042:DBPH:AC:ZnSr=100:1:3:0	0 ZnSr
2	7042:DBPH:AC:ZnSr=100:1:3:0.1	0.1 ZnSr
3	7042:DBPH:AC:ZnSr=100:1:3:0.2	0.2 ZnSr
4	7042:DBPH:AC:ZnSr=100:1:3:0.4	0.4 ZnSr
5	7042:DBPH:AC:ZnSr=100:1:3:0.8	0.8 ZnSr

6	7042:DBPH:AC:ZnSr=100:1:3:1.6	1.6 ZnSr
7	7042:DBPH:ZnSr=100:1:0	0 ZnSr (DBPH)
8	7042:DBPH:ZnSr=100:1:0.1	0.1 ZnSr (DBPH)
9	7042:DBPH:ZnSr=100:1:0.2	0.2 ZnSr (DBPH)
10	7042:DBPH:ZnSr=100:1:0.4	0.4 ZnSr (DBPH)
11	7042:DBPH:ZnSr=100:1:0.8	0.8 ZnSr (DBPH)
12	7042:DBPH:ZnSr=100:1:1.6	1.6 ZnSr (DBPH)
13	7042:AC:ZnSr=100:1:0	0 ZnSr (AC)
14	7042:AC:ZnSr=100:1:0.1	0.1 ZnSr (AC)
15	7042:AC:ZnSr=100:1:0.2	0.2 ZnSr (AC)
16	7042:AC:ZnSr=100:1:0.4	0.4 ZnSr (AC)
17	7042:AC:ZnSr=100:1:0.8	0.8 ZnSr (AC)
18	7042:AC:ZnSr=100:1:1.6	1.6 ZnSr (AC)
19	7042:DBPH:AC:talcum:ZnSr=100:1:3:1:0	0 ZnSr (tal)
20	7042:DBPH:AC:talcum:ZnSr=100:1:3:1:0.1	0.1 ZnSr (tal)
21	7042:DBPH:AC:talcum:ZnSr=100:1:3:1:0.2	0.2 ZnSr (tal)
22	7042:DBPH:AC:talcum:ZnSr=100:1:3:1:0.4	0.4 ZnSr (tal)
23	7042:DBPH:AC:talcum:ZnSr=100:1:3:1:0.8	0.8 ZnSr (tal)
24	7042:DBPH:AC:talcum:ZnSr=100:1:3:1:1.6	1.6 ZnSr (tal)

Sheet preparation: Set the press machine temperature at 130°C, take out 40g haake mixture and put in the mold with size of 16cm × 14cm × 4cm, then compression-molded under vacuumed condition for 5min at a pressure of 5MPa.

Oven foaming: The pressed sheets were put into an oven and heated for 12min at 240°C for foaming and crosslinking. Then the samples were taken out and cooled at room temperature for 24h.

2.3 Characterization

Rubber Process Analyzer (RPA) test: A Rubber Process Analyzer 2000p (Alpha Technologies) was used to measure the crosslinking and foaming behavior of polyethylene mixtures.^{9,10} The viscosity obtained from RPA instrument for cross-linkable polyethylene compounds is an index of melt strength for the melting resin. In this study, the viscosity is also used for simplicity to represent the degree of crosslinking of the crosslinked articles. The test process is as follows: take 2g polyethylene mixture samples, then put the sample into the test wheel, test conditions were maintained at an angle of 0.5° and an oscillation frequency of 100 CPM (1.667 Hz), 230□ last for 10min.

Expansion Ratio Test: The densities of foamed samples, ρ , were determined by deducing the Archimedes law involving weighing polymer foam in water with a sinker using an electronic analytical balance (HANG-PING FA2104), calculated using equation 1.

$$\rho = \frac{a}{a+b-c} \times \rho_w \quad (1)$$

where a, b, and c are the weights of the sample in air without sinker, the totally immersed sinker, and the specimen immersed in water with sinker, respectively, and ρ_w is the density of water. The volume expansion ratio of the crosslinking polyethylene foaming material, R_v , was the ratio of the bulk density of initial mixture density (ρ_0) to that of the crosslinking polyethylene foaming material (ρ), calculated as follows:

$$R_v = \frac{\rho_0}{\rho} \quad (2)$$

Compression strength test: The compression behavior of the cross-linking polyethylene foaming materials were also analyzed using the universal mechanical testing machine (5965, INSTRON Company, American). The compressive tests at room temperature were performed in air. For this purpose, samples with 50cm × 50cm foursquare size were employed. Stress-strain curves were obtained at a rate of 50 mm/min and the samples were compressed to 50% of its original height for according to the Standard ASTM D695-02.

Scanning Electron Microscope (SEM) Test: Those foam samples were immersed in liquid nitrogen for 10min, and then fractured; the surface was vacuum coated with gold examined using a NOVA Nano SEM450 (oxford instruments). The size and size distribution of the cells were measured using Nano-measure software.

Gel content Test: The gel content was determined by using xylene as the solvent according to ASTM-D2765, oil bath temperature was 170°C. A polyethylene sample of 0.400 ± 0.015 grams by weight was cut into small pieces and placed in a 100-mesh stainless steel wire cloth. The sample was dropped into xylene, boiled and reflux condensation for 8 hours. After the extraction cycle, the sample was dried in a vacuum oven at 160°C for 2 hours. The gel content (%) was calculated as follows:

$$X_{gel} = (m_3 - m_1) / (m_2 - m_1) \times 100\% \quad (3)$$

where m_1 is the weight of stainless steel wire cloth, m_2 is the weight of initial sample and stainless steel wire cloth, m_3 is the total weight of sample and stainless steel wire cloth after extraction.

3 Results and Discussion

3.1 Effect of ZnSr on pressure

In this paper, ZnSr was employed as the foaming agent addition to facilitate the AC decomposition, and there have been some reports about the ZnSr as the catalyst in AC decomposition^[14]. In this experiment, Rubber Process Analyzer (RPA) 2000p was used to character the foaming reaction process of AC and ZnSr mixture which inner polyethylene matrix. Fig.1 was the viscosity curve of 0ZnSr and pressure curves of 0ZnSr, 0.1ZnSr, 0.8ZnSr and 1.6ZnSr.

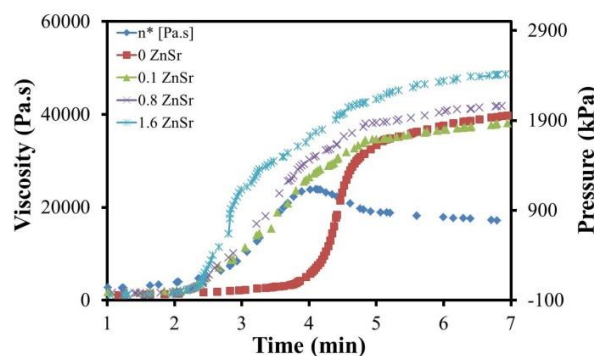


Fig.1 viscosity curve of 0ZnSr and pressure curves of 0ZnSr, 0.1ZnSr, 0.2ZnSr, 0.4ZnSr, 0.8ZnSr and 1.6ZnSr

As shown in Fig.1, the red and the blue curves were the pressure and viscosity changing curves of 0ZnSr, which corresponding to the foaming reaction and crosslinking reaction, respectively. Obviously, when there was no ZnSr in polyethylene mixture, the crosslinking reaction occurred before foaming reaction, and crosslinking reaction began at 3min, and foaming reaction began at 4.3min, the foaming

reaction time lasted for 2.7min. After added 0.1% ZnSr, the foaming reaction started forward to 3min, which started at the same time with crosslinking reaction, and the foaming reaction lasted for 4min. When ZnSr content added to 0.8%, the foaming reaction started at 2.8min, the foaming reaction lasted for 4.2min. Increased ZnSr amount to 1.6% sequentially, the foaming reaction started at 2.5min, which even started earlier than crosslinking reaction, and the foaming reaction lasted for 4.5min. This phenomenon indicated that the more the ZnSr content, the earlier the foaming reaction occurs. And it also means that the presence of ZnSr broadened the foaming window of polyethylene mixture. In addition, there was another phenomenon, that was the final pressure value raised with the increased of ZnSr content, the main reason for this phenomenon was that the foaming reaction was less constrained by the crosslinking reaction when ZnSr content increased and resulted in foaming reaction started earlier than crosslinking reaction^[15,16].

3.2 Effect of ZnSr on viscosity and gel content

According to those above experimental results, we can know that ZnSr could affect the pressure of crosslinking polyethylene foaming material obviously. However, viscosity is another important characteristic of crosslinking polyethylene foaming material. In this experiment, RPA was used to test the viscosity variation with different ZnSr amount, and the test results were showed in Fig.2a.

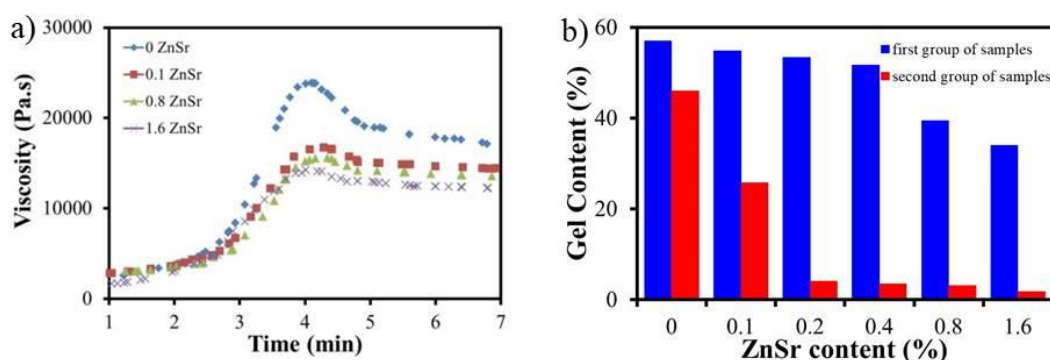


Fig.2 (a)Viscosity variation curves with different ZnSr amount; (b) Gel content of first group of samples and second group of samples with different ZnSr amount

As shown in Fig.2a, when there was no ZnSr in polyethylene mixture, the maximum viscosity was about 25000Pa.s; after adding 0.1% ZnSr, the maximum viscosity decreased to 18000Pa.s; when increased the ZnSr amount to 1.6%, the maximum viscosity decreased to 16000Pa.s continually. These experimental results demonstrated that ZnSr could reduce the crosslinking degree of polyethylene.

In order to verify the effect of ZnSr on crosslinking reaction of polyethylene, gel content was also tested. The experimental results were shown in Fig.2b. The gel content of first group of samples (0 ZnSr, 0.1 ZnSr, 0.2 ZnSr, 0.4 ZnSr, 0.8 ZnSr and 1.6 ZnSr) were shown in Fig.2b. When there was no ZnSr in polyethylene mixture, the gel content was 45.99%; after 0.1% ZnSr was added, the gel content was dropped to 25.81% obviously; when ZnSr amount was increased to 0.2%, the gel content was dropped to 4.15% sharply; when ZnSr amount increased to 0.4%, the gel content was dropped to 3.48%; when ZnSr amount increased to 0.8%, the gel content was dropped to 3.13 % sequentially; and when ZnSr amount increased to 1.6%, the gel content dropped to only 1.83%. With the ZnSr content increased, the gel content was decreased gradually. This experimental result was consisted with the Fig.2 viscosity variation.

In order to avoid the influence of foaming agent AC, the gel content of second group of samples (0 ZnSr (DBPH), 0.1 ZnSr (DBPH), 0.2 ZnSr (DBPH), 0.4 ZnSr (DBPH), 0.8 ZnSr (DBPH) and 1.6 ZnSr (DBPH)) were also tested, which without AC in polyethylene mixture. Test results showed that when there

was no ZnSr, the gel content was high up to 57%; after added 0.1% ZnSr, the gel content decreased to 54.83% slightly; when ZnSr content increased to 0.8%, the gel content decreased to 39.42% obviously; when ZnSr amount increased to 1.6%, the gel content decreased to only 33.97%. This decreasing trend of gel content variation with increasing ZnSr content was similar with the first group of samples, and it demonstrated that ZnSr inhibited the crosslinking reaction^[17]. The main reason for this phenomenon was that ZnSr captured the decomposition peroxide of DBPH and inhibited the process of crosslinking reaction^[18]. In addition, there was another phenomenon that was the gel contents of second group of samples were higher than the first group of samples. This difference demonstrated that foaming agent AC also could inhibit the crosslinking reaction^[19,20].

3.3 Effect of ZnSr on density and expansion ratio

ZnSr not only affect the pressure and viscosity of crosslinking polyethylene foaming materials, but also affect the density and expansion ratio. In this experiment, the density and expansion ratio were tested, and the test results were listed in Fig.3.

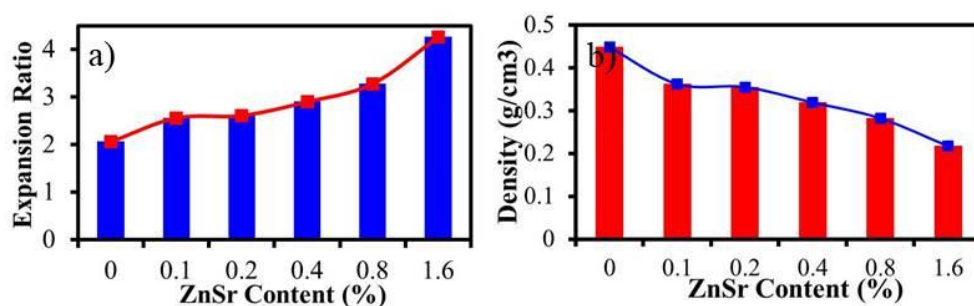


Fig.3 (a) Expansion ratio and (b) density variation with ZnSr content

As shown in Fig.3a, expansion ratios of first group of samples (0ZnSr, 0.1ZnSr, 0.2ZnSr, 0.4ZnSr, 0.8ZnSr and 1.6ZnSr) presented increasing trend with the increased the ZnSr content. When there was no ZnSr, the expansion ratio was only 2.06, after added 0.1% ZnSr, the expansion ratio raised to 2.56; it even raise to 4.26 when the content of ZnSr was up to 1.6%. Oppositely, the density appeared the decreasing trend. When there was no ZnSr, the density was 0.45g/cm³, after added 0.1% ZnSr, the density reduced to 0.36g/cm³; and it fall down to only 0.22g/cm³ when the content of ZnSr was up to 1.6%. These variation trends were similar to the pressure curves (Fig.1).

3.4 Effect of ZnSr on cellular structure

Cellular structure was another important property for crosslinking polyethylene foaming materials. In this experiment, cellular structure was characterized by SEM, Fig.4a, Fig.4b and Fig.4c showed the SEM pictures of 0.2ZnSr, 0.4ZnSr, and 1.6ZnSr, respectively. Cell size was large, the average diameter was about 600μm. And cell shape was irregular and crowd between each other. In order to improve the cell morphology, talcum powder was used as nucleating agent added to the polyethylene mixture. Fig.4d, Fig.4e and Fig.4f showed the SEM pictures of 0.2ZnSr(tal), 0.4ZnSr(tal), and 1.6ZnSr(tal), respectively. The cell became uniform and morphology changed to similar circular structure, and cellular size became small, the average diameters of 0.2ZnSr(tal), 0.4ZnSr(tal) and 1.6ZnSr(tal) were 300μm, 350μm and 450μm, respectively. With the ZnSr content increased, the cellular size appeared the increasing trend, which was similar with the expansion ratio variation trend. In addition, the cell became crowd with the amount of ZnSr increasing. The main reason for this phenomenon was that the foaming reaction preceded by the ZnSr, and cell growth less constricted by crosslinking reaction, finally resulted in the larger cellular

size and more crowding cell space^[16].

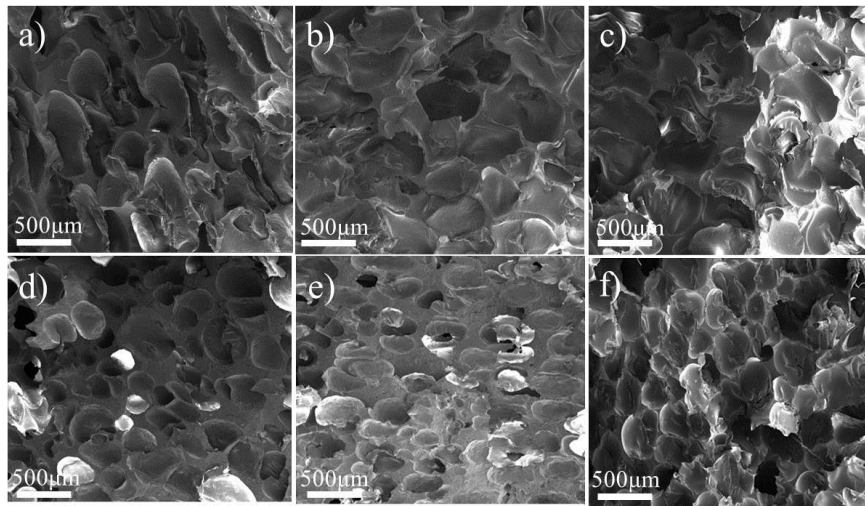


Fig.4 SEM pictures of a) 0.2 ZnSr, b) 0.4 ZnSr, c) 1.6 ZnSr, d) 0.2 ZnSr (tal), e) 0.4 ZnSr (tal) and f) 1.6 ZnSr (tal)

3.5 Effect of ZnSr on compression strength

Due to closely relationship between cellular structure and mechanical property, crosslinking polyethylene foaming materials' mechanical property will be transformed with the cell size and morphology^[21,22], which could affect by different amount of ZnSr. In this paper, compression strength was choose as the typical mechanical property for crosslinking polyethylene foaming material, and different content of ZnSr samples' (first group, third group and four group of samples) compression strength and density were also tested.

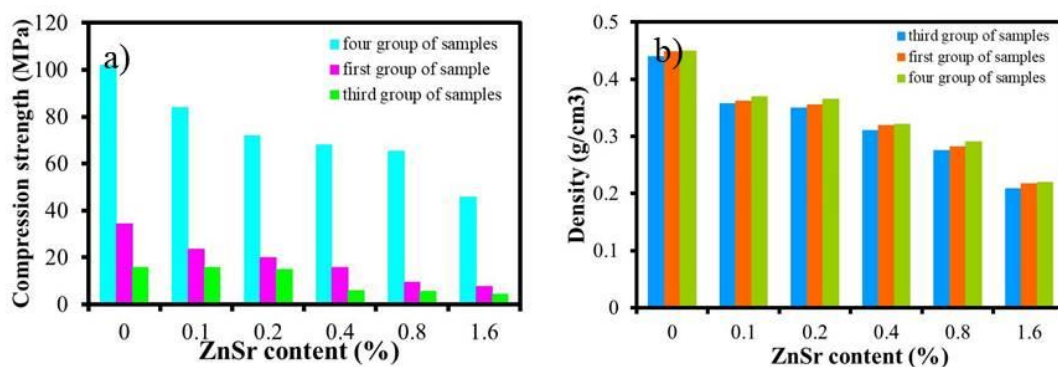


Fig.5 (a) compression strength and (b) density of the first group of samples, the third group of samples and the four group of samples

The compression strength of the first group of samples (0ZnSr, 0.1ZnSr, 0.2ZnSr, 0.4ZnSr, 0.8ZnSr and 1.6ZnSr) showed in Fig.5. Compression strength of 0ZnSr was 34.41MPa, after added 0.1% ZnSr, the

compression strength dropped to 23.55MPa; when the amount of ZnSr increased to 0.2%, the compression strength reduced to 19.83MPa; when ZnSr content increased to 0.4%, the compression strength decreased to 15.68 sequentially; when ZnSr content increased to 0.8%, the compression strength decreased to 9.49MPa; when ZnSr content increased to 1.6%, the compression strength even decreased to 7.64MPa. These above compression strengths presented decreasing trend with ZnSr content increased. This phenomenon was similar with the density variation trend (Fig. 5b), and it demonstrated that: the more content of ZnSr, the lower density, and the lower compression strength.

In order to demonstrate the mechanical property advantage of this controllable crosslinking polyethylene foaming material, the third group of samples were used as the control samples, which didn't have the crosslinking agent DBPH. Fig.5a also showed that the compression strengths of the third group of samples (0ZnSr(AC), 0.1ZnSr(AC), 0.2ZnSr(AC), 0.4ZnSr(AC), 0.8ZnSr(AC) and 1.6ZnSr(AC)). Test results showed that third group of samples' compression strengths lower than the first group of samples, and their compression strengths also presented similar decreasing trend with ZnSr content increased. This phenomenon demonstrated that crosslinking polyethylene foaming material have better mechanical property^[23,24].

Due to the cellular size and morphology have closely relationship with mechanical property, and adding nucleating agent talcum powder, the cellular morphology became more uniform and similar circular structure, and cellular size became smaller, so the compression strengths of mixtures with talcum powder (four group of samples) were also tested in this experiment. Test results showed in Fig.5a, compression strengths of four group of samples were much higher than first group and third group of samples, although these three group of samples with the similar density. This phenomenon main explained by that small cellular size and uniform dispersion foam structure by nucleating agent improved the compression strength^[25].

4 Conclusions

In this paper, the controllable crosslinking polyethylene foaming materials were developed by adding foaming agent addition ZnSr. After added ZnSr, foaming reaction realized starting earlier than crosslinking reaction, the foaming window became wider, density became smaller and expansion ratio became larger. On this basis, nucleating agent talcum powder modified crosslinking polyethylene foaming material owned more uniform cellular structure and higher compression strength.

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