Thermomagnetic formation of single-modulation state in chromium single crystals

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We respond to a critical article by Fawcett *et al.* [Sov. Phys. JETP **67**. No. 3, 379 (1988)] concerning the "inconsistency" of the cryomagnetic (CM) method. Neutron-diffraction measurement data for new crystals and earlier results are used to confirm the viability of the CM method and to compare its effectiveness with that of other methods for various crystal states and after a series of high-temperature anneals. Possible factors influencing the effectiveness of CM cooling are discussed.

It was stated in Ref. 1 that the cryomagnetic (CM) method of producing a single-modulation antiferromagnetic $(1-Q)$ state,[1)] wherein the sample is cooled from room temperature (to that of liquid nitrogen as in Ref. 1) in a field $\mathbf{H}$ (Refs. 4,5) is inconsistent. This conclusion is based on a neutron-diffraction comparison with the initial state of the behavior of three modulation components ($I_x = 31\%$, $I_y = 33\%$, $I_z = 36\%$, $I = 0.89$) of an arc-produced crystal cooled in a field $H = 2.5$ T ($\mathbf{H}\|\mathbf{Q}_z$) through T_N, from room temperature (295 K) to $T < T_{SF}$ (T_{SF} is the spin-flip temperature, $\simeq 123$ K), from 295 K to 148 K (with cooling at 148 K), and from $T > T_N$ to $T < T_{SF}$. The last cooling combined the H_0 (cooling field) and CM methods. The closest approximation to a complete $(1-Q)$ state was obtained by the H_0 method ($I = 0.30$). Less effective was CM cooling to 148 K ($I = 0.55$), and least effective was similar cooling to $T < T_{SF}$ ($I = 0.72$). After (H_0 + CM) treatment, the magnetic state of the crystal remained practically the same as the initial one ($I = 0.88$). These data by Fawcett *et al.* differed greatly from the results of the developers of the CM method,[4,5] who achieved in an iodide crystal by the CM and the H_0 methods $(I-Q)$ states with $I \approx 0.04$ and 0.60, respectively, at approximately the same value of H. The practical equality of the states after (H_0 + CM) treatment to $T < T_{SF}$ to the initial state was attributed in Ref. 1 to the fact that the action of H below T_{SF} restored the volumes of the previously suppressed modulations with $\mathbf{Q}_i \perp \mathbf{H}$, which were preserved when the temperature was subsequently raised through T_{SF} at $H = 0$. That is to say, (H_0 + CM) treatment to $T < T_{SF}$ destroyed in this case completely the magnetic state formed by cooling the sample in a magnetic field through T_N. In our investigations[4,5] we observed no restoration of domains with $\mathbf{Q}_i \perp \mathbf{H}$ after (H_0 + CM) cooling to $T < T_{SF}$ in fields up to 3.2 T.

It should be noted that the developers of the CM method did not postulate the need for cooling the crystal in a field down to $T < T_{SF}$ but, as shown in Ref. 4 and confirmed in Ref. 5, CM cooling in a field 3.0 T is effective only down to 230 K. Further cooling in $\mathbf{H}$ to 77 K had no influence on the level of the formed $(1-Q)$ state (see Fig. 2 or Ref. 4). The cooling to 77 K in some of our experiments was due only to methodological considerations.

After becoming acquainted with Ref. 1, we measured by neutron diffraction, in a iodide Cr–1 crystal ($T_N = 310 \pm 0.2$ K; $T_{SF} = 123 \pm 1$ K; $Q_x > Q_y > Q_z$), similar to that investigated in Ref. 4, the field dependence of the transition to the $(1-Q)$ state under the influence of CM cooling to 140 K at $\mathbf{H}\|\mathbf{Q}_y$. As seen from the data at the end of this paragraph, an approach to a complete $(1-Q)$ state ($I \approx 0.08$) was reached already at $H \approx 1.6$ T. The accuracy of the intensity measurement in our experiments was 6%, i.e., lower than in Ref. 1, but demonstrated fully the viability of the CM method.

H, T	0,08	0,40	0,57	1,00	1,62	2,07	2,60	2,81	3,00	3,20
I	1,10	0,70	0,60	0,38	0,09	0,06	0,07	0,06	0,07	0,09

We performed with the same sample experiments by the method of Fawcett *et al.* With the field (2.5 T) oriented along the vector $\mathbf{Q}_y$, the crystal was subjected to a number of magnetic anneals, after each of which we monitored the intensity of a selected modulation and one of the suppressible ones, and assumed, as in Refs. 1, 4, and 5, that the second suppression of the modulation decreases approximately in proportion to the first. The magnetic state of the crystal at 295 K after various treatment is shown in Table I, from which it follows that: a) the initial magnetic state of the crystal has quite stable anisotropy (row 1); b) the degree of anisotropy increases after cooling the sample from 295 K to 140 K (row 2); c) the efficiency of CM cooling to $T > T_{SF}$ (row 4) or to $T < T_{SF}$ (row 5) exceeds substantially (with respect to the chosen modulation and with respect to the change of the ratio I), the H_0 method just as in Refs. 4 and 5 (row 3); d) comparison of the action of $\mathbf{H}$ at room temperature (row 6) with the influence of CM cooling to 140 K (row 4) demonstrates clearly the efficiency of the latter; e) (H_0 + CM) treatment to $T > T_{SF}$ (row 7) and to $T < T_{SF}$ (row 8) is about (10–12)% more effective than CM cooling (rows 4 and 5, respectively), but this is manifested mainly, as already stated in Ref. 6, at $H > (1.8–2.0)$ T; f) the transition of the crystal temperature through T_{SF} in the course of CM and (H_0 + CM) cooling (rows 5 and 8, respectively) did not lead to a state equivalent to the initial one, i.e., the increase, reported in Ref. 1, of the volumes of modulation with $\mathbf{Q}_i \perp \mathbf{H}$ below T_{SF} under the action of $H = 2.5$ T was not observed (the value of T_{SF} decreased by $\approx 3–4$ K in such a field).

To prove the last statement more rigorously, we monitored the state of the suppressible modulation of Q_x in a field 2.5 T at temperatures above and below T_{SF} against the behavior of the $(\delta,1,0)$ reflection whose intensity is approximately doubled on going from the AF_1 phase to the AF_2 phase. The result was likewise negative.

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TABLE I.

№№	Type of processing	Intensity, %			I_x/I_y
		I_x	I_y	I_z	
1	Initial state	42,0	38,0	20,0	11,1
2	After cooling to	49,0	43,0	8,0	1,14
3	H_0-cooling to	33,0	55,0	12,0 *	0,60
4	CM-cooling to	8,0	85,0	7,0 *	0,09
5	CM-cooling to	8,5	85,0	6,5 *	0,10
6	After applying **H** at	38,0	48,0	14,0 *	0,79
7	$(H_0 + \text{CM})$-cooling to	4,7	90,6	4,7 *	0,05
8	$(H_0 + \text{CM})$-cooling to	3,0	94,0	3,0 *	0,03

*assumed specified values

The higher effectiveness of CM and $(H_0 + \text{CM})$ cooling to $T > T_{SF}$ than of H_0 treatment was noted also when **H** was directed along the wave vectors of the larger and smaller modulations in the initial state. In the case $\mathbf{H} \| \mathbf{Q}_z$ the lower chosen modulation (20%), which was decreased to 8% only by the cooling factor (row 2), was increased by CM treatment to $T > T_{SF}$ almost to the same level as of the modulation of Q_x and Q_y at the corresponding field direction. Nor was restoration of domains with $Q_i \perp \mathbf{H}$ observed following CM and $(H_0 + \text{CM})$ cooling to $T < T_{SF}$ along $\mathbf{Q}_x$ and $\mathbf{Q}_z$.

The single-modulation state formed by CM cooling is quite stable in magnetic fields. This is evidenced by experiments performed by us earlier on the sample investigated in Ref. 4. A 90° rotation of a field $H = 3.0$ T at 130 K away from the direction of the dominant vector $\mathbf{Q}_z$ to the vector $\mathbf{Q}_y$ in a previously produced $(I - Q_z)$ state has practically no effect on the ratio $I = 0.02$, and CM cooling to $T > T_{SF}$ in the same field along the vector $\mathbf{Q}_y$ applied to a sample with previously formed $(1 - Q_z)$ state increases this ratio to 0.10. The high stability of the state produced by CM cooling is indicated also by the fact that its destruction in the sample of Fawcett *et al.* in a magnetic field below T_{SF} turned out to be incomplete (if this is not a consequence of temperature hysteresis).

To obtain additional information on the viability of CM cooling by the scheme of Fawcett *et al.*, we investigated a number of crystals produced by the iodide and arc methods. The iodide crystals, judging from the behavior of the effectiveness of the methods of producing the $(1 - Q)$ state, had the following tentative distribution: in two samples the CM cooling substantially exceeded the H_0 method (analogs of Cr-1). In three samples a state with $I \approx 0.2$ was reached by H_0 treatment while CM cooling had practically no effect (analogs of iodide crystal Cr-3 in Fig. a of our Ref. 7). In the next three samples the H_0 and CM methods were practically ineffective, and only $(H_0 + \text{CM})$ cooling produced an insignificant mutual change of modulation tuning (I changed from 0.90 to 0.70). The values of T_N were 310.5 and 311 K for the crystals of the first group, 308.5, 309, and 312 for the crystals of the second, and 306, 309, and 311 K for the third, i.e., they are all in one temperature interval. Consequently, there is not clear-cut correlation between the value of T_N and the efficiencies of the considered methods.

The results of an investigation of an arc-produced crystal with stable anisotropy (Cr-2) are listed in Table II. A 2.5 T field was directed along the wave vectors of the smaller of the two observed modulations (row 1). CM cooling to $T > T_{SF}$ (row 3) is in this case somewhat less effective than the H_0 method (row 2), while $(H_0 + \text{CM})$ cooling to $T > T_{SF}$ (row 4) and to $T < T_{SF}$ (row 5) exceeds the first two methods. These data on the lower efficiency of CM cooling to $T > T_{FS}$ agree in part with the results of Ref. 1. It must be noted, however, that so low an efficiency of CM cooling to 230 K in an arc-produced crystal was observed already on our earlier investigations[7] (see Fig. 1). It was also reported there that fluctuations of the efficiencies of the H_0 and CM methods were observed for several samples of this type. Figure 1b shows the field dependences, obtained in Ref. 7, of the transition of one such crystal to the $(1 - Q)$ state. We see that the CM cooling to $T > T_{SF}$ exceeds in this case the H_0 method, but $(H_0 + \text{CM})$ processing exceeds the first.

An estimate of the action of CM cooling to 140 K along each of the vectors $\mathbf{Q}_i$ on arc-produced samples with noticeably pronounced anisotropy has shown that the maximum and minimum efficiencies were observed as a rule for **H** directed along $\mathbf{Q}_i$ with larger and smaller modulation in the initial state, respectively. At insignificant anisotropy, the action of the CM cooling in the three direction is also more uniform for both lower and higher efficiency levels than in the H_0 method. No such analogs of the Cr-1 sample were observed among the arc-produced crystals, and $(H_0 + \text{CM})$ cooling to $T < T_{SF}$ produced no state equivalent to the initial one.

TABLE II.

№№	Type of processing	Intensity, %		I_x/I_y
		I_x	I_y	
1	Initial state	57	43	1,34
2	H_0-cooling to 295 K	48	52	0,92
3	CM-cooling to 140 K	52	48	1,08
4	$(H_0 + \text{CM})$-cooling to 140 K	31	69	0,45
5	$(H_0 + \text{CM})$-cooling to 115 K	30	70	0,43

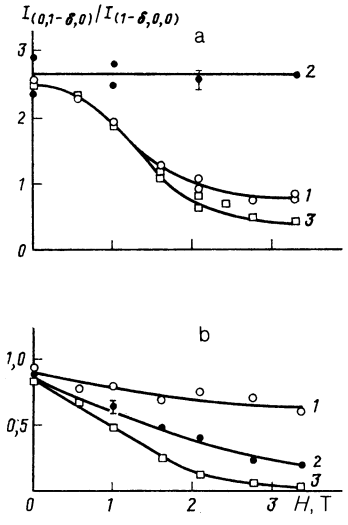

FIG. 1. Field dependences of transition of iodide (a) and arc-generated (b) crystal to the $(1-Q)$ state. 1, 2, 3—H_0, CM, and $(H_0 + \text{CM})$ treatments, respectively, $\mathbf{H}\|\mathbf{Q}_x$.

Experimental data exist therefore attesting to higher (our) and lower (our, of Fawcett *et al.*) efficiency of CM cooling than by the H_0 method, as well as data indicating that the $(1-Q)$ state is destroyed below (Fawcett *et al.*) T_{SF} by $\mathbf{H}$ or remain unchanged (our data). The discrepancy between these results is undoubtedly due to structure peculiarities in the investigated crystals of different origin.

The assumption that anisotropy is responsible for the high efficiency of the CM method must apparently be refuted, since both high and low viability of this method are observed at approximately the same anisotropy level (high or low). Such a comparison seems to eliminate also the anisotropy as a cause of the nonrecovery, in our crystals, of domains with $\mathbf{Q}_i \perp \mathbf{H}$ at $T < T_{SF}$ (Ref. 1).

High or low efficiency of the CM cooling method can be attributed to the influence of one or several impurity elements that accelerate or hinder the rotation of the vectors $\mathbf{Q}_i$ along $\mathbf{H}$ above T_{SF} and stabilize or weaken their positions below T_{SF}. However, analysis of the impurity content of iodide or arc crystals (if we are not misled by mass-spectroscopy data from the surface) and of their T_N values makes such a choice impossible so far.

To identify the causes of the different efficiency of CM cooling of crystals of seemingly the same type,and to come closer to the experimental results of Fawcett *et al.*, a number of high-temperature anneals were made on the arc-produced Cr-3 crystal ($T_N = 311$ K; $Q_x : Q_y : Q_z = 26 : 29 : 45$), on which the H_0 and CM methods hardly acted ($H = 3$ T and $\mathbf{H}\|\mathbf{Q}_x$) and the efficiency of $(H_0 + \text{CM})$ cooling to 140 K was negligible (Table III, column 1). Annealing at 1000 and 1200 °C (vacuum $1 \cdot 10^{-5}$ Torr) resulted in lower values of I than in the initial state (column 1) and consequently in a change of the anisotropy level, a decrease of the temperature hysteresis (ΔI^g), and an increase of the efficiencies of all methods,with substantial predominance of CM and $(H_0 + \text{CM})$ cooling. Raising the annealing temperature of 1400 °C (Ar atmosphere) increased insignificantly the selected modulation (row 1), had practically no influence on the efficiency of the H_0 method, and lowered the viabilities of CM and $(H_0 + \text{CM})$ coolings. A subsequent 72-h annealing at 1200 °C (vacuum $\approx 1 \cdot 10^{-5}$ Torr) and cooling to room temperature at $\approx 50°$/h raised greatly the efficiency of the H_0 method, almost to the level of the $(H_0 + \text{CM})$ method, and annihilated almost completely the viability of the CM cooling. The efficiency of the $(H_0 + \text{CM})$ treatment was then determined in practice by the viability of the H_0 method. To estimate the influence of the rate of cooling from high temperature on the behavior of the modulations under the treatments, the sample was reheated to 1200 °C and then cooled at a rate 2400°/h. As seen from the last column, the state of the crystal was the same as after the preceding anneal.

The behavior of the efficiencies of the H_0, CM, and $(H_0 + \text{CM})$ methods used for another arc-produced sample (Cr-4) with initial parameters similar to those of Cr-3, after annealing at 1100 °C (vacuum) was approximately the same as for the Cr-3 crystal after annealing at 1200 °C. However, the great increase of the efficiency of the H_0 method, practically to the level of the $(H_0 + \text{CM})$ cooling,and the complete loss of viability of the CM method in this case occurred already after 2 h of annealing at 1400 °C (Ar).

The influence of the anneals on the efficiencies of the considered methods was estimated also for an iodide Cr-5 sample having likewise a rather abrupt anisotropy and a stable temperature hysteresis (Table IV, rows 1 and 2 respectively). In the initial state the CM method was practically blocked, while the H_0 and $(H_0 + \text{CM})$ treatments produced states with $I \approx 0.79$ and 0.41 respectively (column 1). After annealing at 1200 °C (vacuum) the efficiencies of the last two methods increased practically to the maximum (for the given $H = 3$ T), but after raising the annealing temperature to 1400 °C (Ar) it returned unexpectedly to the initial level (column 1). What is decisive in the behavior of $(H_0 + \text{CM})$

TABLE III.

№№	Type of processing	I_y/I_x					
		Start	1000° C, 1 h	1200° C, 2 h	1400° C, 2 h	1200° C, 72 h	1200° C, 2 h
1	Initial state	1,12	0,91	0,90	0,85	0,77	0,78
2	Cooling to 140 K	2,11	1,17	0,82	0,90	0,80	0,80
3	H_0-cooling to 295 K	1,03	0,90	0,65	0,66	0,16	0,15
4	CM-cooling to 140 K	1,04	0,58	0,24	0,40	0,76	0,76
5	$(H_0+\text{CM})$-cooling to 115 K	0,72	0,20	0,04	0,10	0,09	0,11

TABLE IV.

№№	Type of processing	Ratio I_x/I_y $H \parallel Q_y$		
		исходное	1200° C	1400° C
1	Initial state	2,20	2,27	**1,32**
2	Cooling to 140 K	2,89	2,55	**1,80**
3	H_0-cooling to 295 K	0,79	0,11	**0,78**
4	(CM-cooling to 140 K	2,62	2,63	**1,25**
5	$(H_0 + \text{CM})$−cooling to 115 K	0,41	0,07	**0,47**

cooling after all the anneals is the efficiency of the H_0 method, while the CM treatment is affected only by changes in the anisotropy and in the value of ΔI^g (rows 1 and 2, respectively). Evidently, it was impossible to restore the viability of the CM method by annealing.

The results of the last three experiments have shown the following: a) anneals did not change our samples to a state at which, as in Ref. 1, domains with $\mathbf{Q}_i \perp \mathbf{H}$ are restored below T_{SF} in a field (2.5–3.0) T; b) increasing the annealing duration (or temperature) produced a region of states with high CM-cooling efficiency and low viability of the H_0 method, but followed by a region in which the H_0 method reaches a maximum efficiency and the CM cooling is no longer viable. The closest approach to a complete $(1 - Q)$ state is reached in the former case as a result of $(H_0 + \text{CM})$ cooling. c) In the course of annealing the iodide crystal the region of states with high efficiency of the H_0 method was followed by a state in which the viability of this method is also lost.

The increased efficiencies of all the methods following an annealing-temperature rise to (1100–1200)°C is in full accord with the notion of uniform lowering of the level of the internal stresses in arc-produced crystals. This explains also the subsequent increase of the H_0-method efficiency when the temperature is raised to 1400 °C (or the duration of annealing at 1200 °C is increased) for samples of the same type, and also the increased efficiency of this method for the iodide crystal after annealing at 1200 °C. It is difficult, however, to interpret the decrease of the CM-cooling efficiency when the temperature is raised to 1400 °C (or the exposure at 1200 °C is lengthened) for an arc-produced sample, the total blocking of the CM method after all the anneals, and the deterioration of the viability of the H_0 method after annealing an iodide crystal at 1400 °C. A "simple" annealing in this case can be the assumption of possible contamination of the sample under the annealing conditions. In this case, in our opinion, the fastest to decrease should be the efficiency of the CM cooling which is most sensitive to interstitial impurities, followed by the H_0 method. If, however, the sample-surface mass-spectrometer analysis data do not mislead us, the impurity content of the samples was insignificantly changed. It can also be premised that annealing produces in the sample a redistribution of various defects that act selectively either on the rotation of the $\mathbf{Q}_i$ vectors below room temperature, or on the formation of domains with $\mathbf{Q}_i \parallel \mathbf{H}$ at the point T_N. The redistribution of the defects can obviously be accompanied by entry of interstitial elements from the annealing ambient.

The CM cooling process consists of two factors (temperature-governed changes including hysteresis, and the action of **H**), the influence of which depends on the state of the crystal structure. In samples with an internal-stress level that is on the whole small (and in the presence of anisotropy), the predominant action, in our opinion, is that of the field. In this case the CM cooling forms at low values of **H**, with approximately equal success, an $(1 - Q)$ state with a wave vector having larger as well as smaller modulation in the initial state. For samples with this state of the structure (type Cr-1) the CM cooling is substantially more effective than the H_0 method. In more stressed mainly arc-produced crystals (usually with strong anisotropy) the temperature changes with ΔI^g are decisive. In the case of CM cooling along the larger-modulation vector, both factors, tending to increase the volume of the chosen modulation, also ensure a closer approach to the $(1 - Q)$ state than the H_0 method. $(H_0 + \text{CM})$ treatment leads at the same H direction to a state with I (0.30–0.10). In the case of CM cooling with **H** along the vector $\mathbf{Q}_i$ at a much smaller modulation, temperature variation of (ΔI^g) the decrease of this modulation is much larger and the field is not always capable of compensating for it. In the upshot, in CM treatment the smaller modulation is not only incapable of increasing compared with the initial value, but even fails to reach the proper level. That is to say, the CM cooling counteracts in this case the creation of an $(1 - Q)$ state. The advantage of the H_0 method at this orientation of **H** is obvious, and the $(H_0 + \text{CM})$ treatment has an efficiency intermediate between those of the H_0 and CM methods.

The foregoing comparison and the material set forth above demonstrate unambiguously that a) CM cooling, contrary to the assumption based on our earlier measurements,[4,5] is not more effective than the H_0 method for all structural states of the crystals, but only for some selected ones (as is incidentally also the H_0 method), and there are therefore no grounds for considering it as inconsistent;[1] b) when a method is chosen for a successful formation of an $(1 - Q)$ state it is necessary to have an idea of the prior history of the crystal and of the behavior of this modulation components with change of temperature.

The nature of the CM effect has not yet been determined, the causes of the discrepancies between our and Fawcett's results[8] are not clear, nor has the state in which the CM cooling efficiency is a maximum been established uniquely. It seems to us that this state is governed by the special form of the relation between the structure defects and the magnetostriction effects, a relation that ensures the maximum weakening of the vectors $\mathbf{Q}_i$ by the action of **H** in the interval in which, on decrease from room temperature, the temperature difference between the lattice parameters first increases to the maximum (270 K) along the vector $\mathbf{Q}_i$ and along the free axis, and then drops to zero at 230 K (Ref. 9) (where the CM method is effective). The question of the type of structure defects and their number is as yet open.

It is not at all surprising that in our experiments below

T_{SF}, in a field (2.5–3.0) T, no reconstruction of domains with $\mathbf{Q}_i \perp \mathbf{H}$ was observed, since for the samples of Werner *et al.*[10] the threshold field was ≈ 11 T, and in Ref. 9 the first appearance of domains with $\mathbf{Q}_i \perp \mathbf{H}$ was noted in fields ≈ 4.5 T. Only Fawcett *et al.*, apparently as a result of some peculiarities of the structure of their crystal, succeeded in obtaining already at $\mathbf{H} = 2.5$ T an almost 100% reconstruction of the previously suppressed modulations.

The causes of the high efficiency of CM cooling to 230 K and of the anchoring of the vectors $\mathbf{Q}_i$ below T_{SF} in certain crystal states calls for additional research preceded by a set of anneals under conditions that prevent sample contamination, and using crystals with near-ideal structure, which constitute 1/500 of the encountered iodide crystals.[11]

We present some details in our experiments. The field was produced by an electromagnet. The background field, 0.08 T, did not affect the state of the magnetic structure at the phase-transition points. The sample was raised above room temperature by heated air, and was cooled by nitrogen vapor. The latter, as verified by special experiments, did not influence the investigated CM effects. After **H** was turned off above or below T_{SF} the sample was allowed to reach, under natural conditions, the room temperature at which the reflection intensities were measured.

[1)] The $(1-Q)$ state was first formed in the course of cooling the crystal through the point T_N in a magnetic field (**H**) directed along one of the principal crystallographic axes (H_0-method).[2] The transition of the sample into the $(1-Q)$ state is determined at room temperature and at $H = 0$ by neutron diffraction, using the field dependence of the ratio of the intensity of a reflection of type $(1-\delta,0,0)$ of one of the suppressed modulations ($\mathbf{Q}_i \perp \mathbf{H}$) to the intensity of the corresponding reflection of the selected ($\mathbf{Q}_i \| \mathbf{H}$) modulations ($I = I_{sup}/I_{sel}$). The degree of perfection of the $(1-Q)$ state was estimated in Ref. 1 from the quantity $R = 2I_z/(I_x + I_y)$, where $R = 1/I$ according to their data. For an ideal $(3-Q)$ state $I_x = I_y = I_z = 33\%$) the result was $I = R = 1$, while for an ideal $(1-Q)$ state, e.g., $I_z = 100$, $I_x = I_y = 0$) it was assumed that $I = 0$ and $R = \infty$. In the present article we estimate the perfection of the $(1-Q)$ state by the ratio I. It is proposed to take the complete $(1-Q)$ to be the state in which $I \leqslant 0.08$.

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