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Development and saturation investigation of MALO saturable absorber

Streszczenie. *Artykuł przedstawia badania efektu nasycania w nowo opracowanym nasycalnym absorberze $MgAl_2O_4$:Co. Absorber został opracowany z myślą o jego termicznym połączeniu z ośrodkiem aktywnym takim jak szkło erbowe. Na podstawie tych badań zostały wyznaczone podstawowe parametry nasycalnego absorbera (z punktu widzenia generacji laserowej) takie jak absorpcyjny przekrój czynny z poziomu podstawowego, absorpcyjny przekrój czynny z poziomu wzbudzonego oraz straty dysypacyjne. Przedstawiono porównanie wyznaczonych parametrów z parametrami przedstawianymi w literaturze stwierdzając pewną przewagę badanego nasycalnego absorbera nad dostępnymi komercyjnie (**Opracowanie i badania efektu nasycania nasycalnego absorbera MALO**).*

Abstract. *The paper describes the investigation of saturation effect in newly developed $MgAl_2O_4$:Co saturable absorber with the aim of thermally bounding it with the active media such as erbium glasses. On the basis of the experimental results the most important parameters, from the point of view of laser generation, such as ground state absorption cross section, excited state absorption cross section and dissipative losses were calculated. The comparison of this parameters with the parameters presented in the literature was done showing the advantages of the newly developed saturable absorber over commercially available ones.*

Słowa kluczowe: nasycalny absorber, absorpcyjny przekrój czynny z poziomu podstawowego, promieniowanie „bezpieczne dla oka".
Keywords: saturable absorber, ground state absorption cross section, "eye-safe" radiation.

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Introduction

In recent years a lot of effort was put into developing new microchip pulsed lasers with output peak power of several kW and generating wavelength in the "eye-safe" range of 1.5 µm. As a result of that there are many papers dealing with different active media and saturable absorbers meant for „eye-safe" microchip lasers [1-15]. However there is still lack of effective microchip laser of this kind. To overcome this lack scientists from the Military University of Technology and Institute of Electronic Materials Technology teamed up to develop thermally bonded (TB) microchip laser consisting of $MgAl_2O_4$:Co (MALO) as a q-switch and glass:Er,Yb as an active medium. Such construction will significantly increase the maximum possible peak power that can be generated by such microchip not damaging it. For this purpose a new MALO crystal doped with cobalt ions as well as glass doped with erbium and ytterbium ions were developed.

The aim of this work is to investigate MALO from the point of saturation and calculate its main parameters such as ground state absorption cross section (GSA) and dissipative losses. Knowing these parameters is essential from the point of view of laser generation as well as TB. The calculation of the main parameters of MALO was done according to the method presented in paper [16]. The work is a some kind of continuation of the results presented in paper [17].

Groth of MALO

$MgAl_2O_4$ single crystals were grown by classical Czochralski Method at the Institute of Electronic Materials Technology (ITME), Warsaw, Poland. First (undoped) MALO was grown on iridium wire. By X-ray technique it was evidenced that it was grown in [111] direction. XRD patterns (see Fig 1 in Ref. [17]) have shown that there were no admixtures of higher order spinels. Namely**,** neither $MgAl_4O_7$ nor $MgAl_6O_{10}$, as well as neither α-Al_2O_3, nor MgO, could be evidenced by the mentioned powder technique. Since the single crystal was achieved, this became later the source of seeds for next processes.

Co-doped crystals were pulled from 50 mm diameter Iridium crucible in Cyberstar Oxypuller arrangement. We used an important earlier information on a distinguished problem, namely of a relatively small yield while cutting the boules into wafers [18]. Possibly, these were the excessive residual stresses that might have been a problem in their experiments. Therefore, we decided to grow 1" diameter crystals only, and to thermally isolate the reaction chamber to the "greatest possible extent", just to avoid thermal losses and to lower the temperature gradients on the liquid-solid interface and in the crystals themselves. Reduction of diameter aims at reduction of residual stresses. So called resolved shear stress (RSS) is directly proportional to the square of the diameter in Czochralski-grown crystals, i.e. the maximal value of RSS in 1" crystals is 4 times lower compared to 2" crystals.

From ITME's own experience in growing various single crystals, including also semiconductors like e.g. Si, GaAs or InP, it follows that reduction of this stress by different methods avoids an earlier problem of cracking, usually expanding from bottom to top of the crystals (in Czochralski grown crystals existing simulations and measurements show that stresses (and disclocation densities) are larger in the bottom parts, compared to top parts (close to the cone and seed). Thanks to the mentioned reduction in diameter, we have never experienced any problem of cracking. Besides, although in the current stage of investigations neither experiments of mechanical strength of the crystals have been yet made, an indirect proof for high structural quality of these crystals might be one of thermal experiments in which a sample heated to 780^{o}C survived, while its holder was incidentally crushed.

So, after limiting the diameter, we decided to use active after-heater, which is an important part of the reactor, because it helps also to limit temperature gradients in these parts of crystals that have already emerged from the liquid (cone and middle part). The active after-heater is also very helpful in limiting temperature gradients in finally extracted from the melt crystals, which, by all means undergo then a certain thermal shock.

MgO, Al_2O_3 and Co_3O_4 high-purity powders (at least 4.5 N) were pressed on the isostatic press and small portions of the pressed powder were added to the crucible, and after a couple of consecutive meltings of these powders, the final melting was ended by inserting MALO seed into the melt. We begun with the lowest (0.06) and by adding suitable quantities of Co_3O_4 after each process, we continued until the largest Co concentration (0.6 at. % in the melt) was reached. Before each processes, the air was pumped away from the reactor, and after that it was exchanged by a high purity N_2.

However, we need now to comment on Co concentration in the crystal. Sorry to say, but the so called segregation (or distribution) coefficient (k_{eff}) of Co in MALO has not been yet determined, or at least published in the world-wide literature. The corrections of Co_3O_4 in the melt in the consecutive processes were made on the assumption that k_{eff} is equal to 1.0. This, theoretically, is an ideal case in Czochralski-growth crystals, and it means that Co distribution is (or should be) homogeneous over the entire grown crystal, as well as it should be (almost) the same as that in the (crystallized) melt after the crystal had been extracted from it. Our assumption that k_{eff} = 1 has, or precisely speaking – had, some indirect proofs in others' experiments. Jouni et al. [19] measured that Mn^{2+} ion in MALO is uniformly distributed within the crystal, as well as k_{eff} = 1 ± 0.1. According to Shannon [20] ionic radii of 2+ ions in tetrahedral positions are as follows : Mn^{2+} 0.66; Co^{2+} 0.58, while this of Ni^{2+} is either 0.55 or 0.49, respectively. They have also measured k_{eff} for Ni^{2+} in MALO to be 0.73 [21], as well as they also observed an uniform distribution of this ion along the crystal axis.

There is no room for further comments on this problem in this paper, which, basically is devoted to investigation of nonlinear absorption. However, in short, k_{eff} strongly depends on ionic radius of the dopant in every crystal. Ni^{2+} ionic radius noticeably departs from this of Mn^{2+}, which exhibits k_{eff} = 1, than that of Co^{2+}. Therefore, one may presume that k_{eff} of Co^{2+} in MALO will be somewhere between 0.73 and 1.0, and, possibly, this will not be faraway departing from unity.

However, practical measurements (especially Flame Atomic Absorption Spectroscopy) have shown that this is not really so. We do not show our figures here, since this phenomenon has still been investigated. Nevertheless, this what needs to be said at the moment is that real concentrations of Co in these crystals are ab. 4-5 times smaller than those predicted on the assumption that k_{eff} = 1. Besides, it was also discovered that Co concentration in the top (conical) parts of the crystals is much larger (ab. 30 – 40 %) than in the adequate bottom parts. This last feature was also confirmed by optical absorption measurements (this paper). Namely, it was discovered that adequate lower quantities of the absorption coefficient associated with Co ions were found in the tail parts of the crystals. In the future by plotting the values of all absorption coefficients vs. real concentrations of Co in MALO we will be able to discover the true value of the absorption cross-section (σ_N) , a very important parameter in laser technique. σ_N reported in the literature (2.1 [22] and 1.2 [23] ($x10^{-19}$ cm^2) for 540 and 1540 nm, respectively) seem to be highly uncertain. In conclusion we may say that Co concentrations reported in this paper will be that of this concentration in the melt.

Pulling rate was kept between 1.8 and 2.2 mm/h, while rotation rate was varied between 12 and 18 (usually 15) rpm. After automatic extraction of a crystal from the melt a 24 hours cooling time was usually applied.

Standard procedure in sample preparation was to cut off the conical and tail parts perpendicularly to the growth direction and polish the two end faces for optical testing. This (plane and circular polariscopes, spectro-conoscope) revealed a good optical homogeneity of the crystals [17]. A typical as-grown MALO (in the center) and two other boules prepared for testing of their optical (in)homogeneity are shown in Fig. 1. After such examination the boules were later cut into pieces : thin (ab. 1.5 mm) wafers that were usually cut perpendicularly to [111], for laser experiments described in this paper.

Fig. 1. An exemplary two Co-doped MALO boules prepared for testing : two pairs (end faces and on the perimeter, respectively) of surfaces have been polished for optical testing. In the center one can see "as grown" boule (just after pulling).

Spectroscopic and saturation investigation

Six different samples of MALO with atomic concentration of Co 0.085% and 0.16% were examined. The samples were polished but they were not coated. Thus to appropriate calculate their parameters multiple reflection inside the samples had to be taken into account. Absorption coefficient spectra of the samples were calculated on the basis of measured transmission as a function of wavelength. The curves of the absorption coefficient for the investigated samples are presented in Fig. 2.

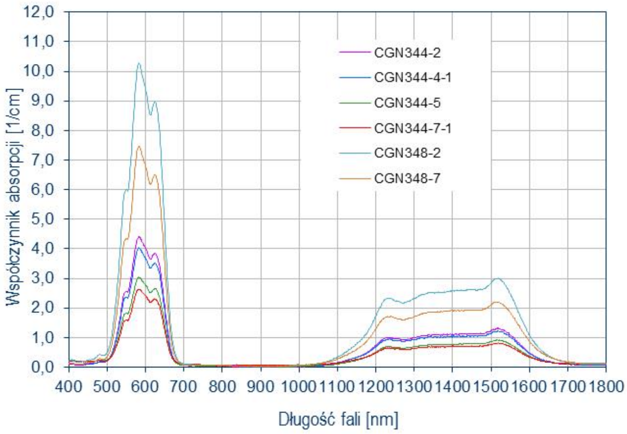

Fig. 2. Absorption coefficient of the investigated samples as a function of wavelength

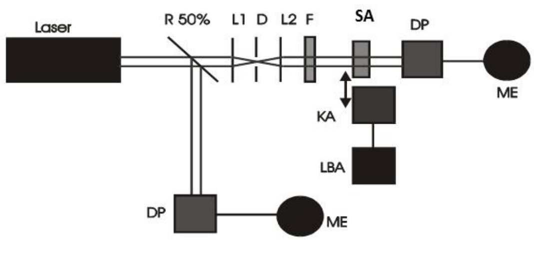

Fig. 3. Experimental setup for investigation of absorber saturation ME - radiometer, DP - energy probe, F - filter, L1 and L2 - lenses, D - diaphragm, SA – saturable absorber, LBA - laser beam analyzer, KA – camera

Saturation investigation of the samples were carried out at 1332 nm and 1535 nm wavelength. For the analysis the Avizonis-Grotbeck's model and the procedure described in paper [16] was used. The small signal transmission of the absorber T_0 and the transmission of the saturable absorber close to but outside the laser wavelength T_λ were calculated from T_{0m} and $T_{\lambda m}$ which are the small signal transmission measured by the spectrometer at the investigated wavelength and the small signal transmission in the region close to but outside the investigated wavelength (2000 nm), respectively. Moreover for each of the samples refractive index n and the fluorescence lifetime t_f was measured. The parameters of the samples as well as the input parameters for the analysis by Avizonis-Grotbeck's model are presented in table 1.

Table 1. Parameters of MALO as well as the input parameters for the analysis by Avizonis-Grotbeck's model

Sample	Concentration of Co at. [%]	Wavelength [nm]	T_{0m} [%]	n	Thickness d [mm]	$T_{\lambda m}$ [%]	t_f [ns]
CGN344-2	0.085	1332	75.5	1.698	1.390	85.72	350
		1535	73.4	1.695			
CGN344-4-1	0.085	1332	78.8	1.698	1.097	87.30	
		1535	77.0	1.695			
CGN344-5	0.085	1332	78.5	1.698	1.435	86.93	
		1535	75.8	1.695			
CGN344-7-1	0.085	1332	80.0	1.698	1.415	86.00	
		1535	77.6	1.695			
CGN348-2	0.16	1332	61.7	1.698	1.414	85.83	
		1535	57.9	1.695			
CGN348-7	0.16	1332	67.9	1.698	1.440	84.24	
		1535	64.4	1.695			

Table 2. Laser beam parameters

Wavelength [nm]	Repetition rate [Hz]	Pulse length [ns]	Pulse energy [mJ]	Waist diameter [mm]	Beam quality M^2	polarization
1332	10	4	4.10	1	1.25	linear
1535	10	4	3.20	1	1.25	linear

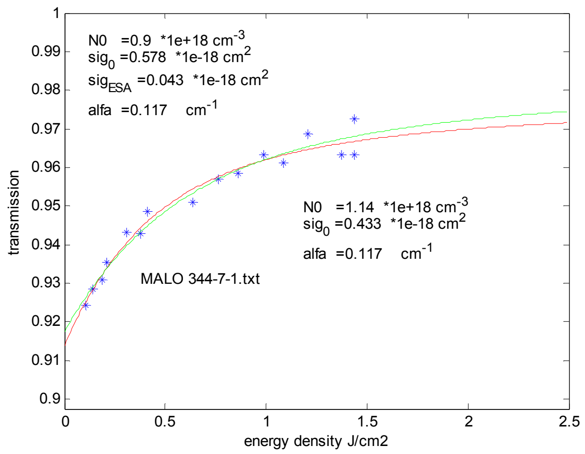

Fig. 4. Exemplary aproximations for CGN344-7-1 samples at 1332 nm wavelenght

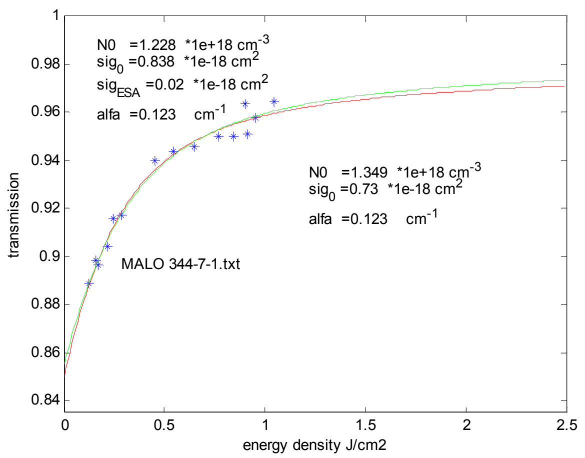

Fig. 5. Exemplary aproximations for CGN344-7-1 samples at 1535 nm wavelenght

Investigations of saturation of the absorbers were carried out using the experimental setup shown in Fig. 3. As a sources of radiation two solid-state lasers were used generating at 1332 nm and 1535 nm. The lasers operated in active gain modulation mode and generated laser pulses of 4 ns length at half-height with repetition rate of 10 Hz. The lasers beams were linearly polarized and characterized by Gaussian distribution. The waist diameters of the beams were equal to 1 mm. The parameters of the laser beams are shown in table 2.

To avoid any uncertainty connected with the laser power fluctuation a plate with 50% transmission and 50 % reflection was used to measure energy of the same pulse before and after the sample. During the investigation the saturable absorbers were placed at the beam waist. To change the energy density filters with different transmission were used. The laser beam diameter on the surface of the saturable absorber was measured by a laser beam analyzer. From the measured beam parameters a local energy density was calculated. The transmission of the investigated saturable absorbers was calculated as a ratio of the transmitted energy to the incident energy. The transmitted as well as incident energy was estimated as an average from 100 repeated measurements. On the basis of the measured transmission the real transmission was calculated taking into consideration multiple reflections inside the samples.

All the investigated samples expressed a very good saturation effect for both laser wavelengths. The exemplary aproximations for one of the samples at 1332 nm and 1535 nm wavelenght are presented in figure 4 and 5, respectivelly. Red color means that the excited state absorption (ESA) was taken into account while green color means that the ESA was passed over. The calculated parameters of the samples (GSA, ESA, concentration of Co - N_0, disipative losses – L) are presented in table 3 and 4. Comparison of the calculated GSA and ESA with the data from literature is presented in table 5. One can see that GSA of the newly developed saturable absorber is higher than that of presented in the literature and commercially available [30] which is highly advantageous to laser pulse generation. It was also shown that the ESA is a little higher than that shown in literature however following the results achieved by Shcherbitsky {28] we should assume that ESA doesn't exist which makes the advantage of our absorber over the other ones even more vivid.

Absorption coefficient k can be expressed as product of concentration and absorption cross section. Thus knowing the ground state absorption cross section for MALO at 1332 nm and 1535 nm wavelength and the absorption coefficients for the same wavelength the concentration can be calculated. The concentration of Co N_{ok} calculated in this way is presented in table 3 and 4.

Tab. 3. Calculated parameters of the samples by Avizonis-Grotbeck'a model at 1332 nm wavelength

		GSA [cm^2] x 10^{-19}	ESA [cm^2] x 10^{-20}	N_0 [cm^3] x 10^{18}	L [cm^{-1}]	k [1/cm]	N_{ok} [cm^{-3}] x 10^{18}
CGN344-2	with ESA	5.22	4.2	2.094	0.143	1.076	2.061
	no ESA	3.88	-	2.675			2.773
CGN344-4-1	with ESA	2.50	1.5	3.75	0.016	1.007	4.028
	no ESA	2.18	-	4.242			4.619
CGN344-5	with ESA	4.53	1.4	1.732	0.041	0,739	1.631
	no ESA	4.12	-	1.876			1.793
CGN344-7-1	with ESA	5.78	4.3	0.9	0.117	0.677	1.171
	no ESA	4.33	-	1.14			1.563
CGN348-2	with ESA	7.06	13.4	4.159	0.131	2.497	3.536
	no ESA	3.01	-	8.32			8.295
CGN348-7	with ESA	3.75	0.3	4.778	0.258	1.838	4.901
	no ESA	3.66	-	4.881			5.021
Average	with ESA	**4.81**	**4.18**	**2.902**	**0.118**	**1.306**	2.716
	no ESA	**3.53**	**-**	**3.856**			3.698

Tab. 4. Calculated parameters of the samples by Avizonis-Grotbeck'a model at 1535 nm wavelength

		GSA [cm^2] x 10^{-19}	ESA [cm^2] x 10^{-20}	N_0 [cm^3] x 10^{18}	L [cm^{-1}]	k [1/cm]	N_{ok} [cm^{-3}] x 10^{18}
CGN344-2	with ESA	6.4	2.8	2.199	0.149	1.226	1.916
	no ESA	5.46	-	2.492			2.245
CGN344-4-1	with ESA	5.4	8.0	2.304	0.024	1.143	2.117
	no ESA	3.34	-	3.465			3.422
CGN344-5	with ESA	5.95	1.2	1.749	0.047	0.869	1.461
	no ESA	5.49	-	1.857			1.583
CGN344-7-1	with ESA	8.38	2.0	1.228	0.123	0.766	0.914
	no ESA	7.3	-	1.349			1.049
CGN348-2	with ESA	6.62	14.6	5.168	0.137	2.809	4.243
	no ESA	2.92	-	10.291			9.620
CGN348-7	with ESA	8.26	9.3	2.791	0.264	2.065	2.500
	no ESA	4.86	-	4.2			4.249
Average	with ESA	**6.83**	**6.3**	**2.573**	**0.124**	**1.480**	**2.165**
	no ESA	**4.89**		**3.942**			**3.023**

Tab. 5. Comparison of the calculated GSA and ESA with the data from literature

literature	GSA [cm^2] x 10^{-19}		ESA[cm^2] x 10^{-20}	
wavelength [μm]	1.3	1.5	1.3	1.5
[24]	4	5.1	3.6	4.6
[25]	2.8	3.5	2.0	1.0
[26]	-	2.8	-	4.8
[27]	2.8	3.5	1.96	1.05
[28]	-	2.4 2.9	-	0 0
[29]	0.25	-	0.38	-
[30]	-	3.5	-	-
Avizonis-Grotbeck'a **with ESA**	**4.81**	**6.83**	**4.18**	**6.3**
Avizonis-Grotbeck'a **no ESA**	**3.53**	**4.89**	**-**	-

Conclusion

The paper presents the results of investigation of saturation effect in newly developed $MgAl_2O_4$:Co saturable absorber. On the basis of that its main parameters such as ground state absorption cross section and dissipative losses were calculated. One can see that the parameters of the newly developed saturable absorber makes it be more desired by laser designers in comparison with the ones presented in the literature and commercially available.

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